DEPARTMENT OF DEFENSE
QUALITY SYSTEMS MANUAL FOR ENVIRONMENTAL LABORATORIES

Prepared By

DoD Environmental Data Quality Workgroup
Department of Navy, Lead Service
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Quality Systems Manual for Environmental Laboratories

VERSION 2 DRAFT

Based On
National Environmental Laboratory Accreditation Program (NELAP)
Chapter 5 (Quality Systems)
NELAP Voted Version 14 – 29 June 2000

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PREFACE TO THE DoD QUALITY SYSTEMS MANUAL

Purpose

The purpose of this document is to provide implementation guidance on the establishment and management of quality systems for environmental testing laboratories that intend to perform work for DoD. This guidance is based on the National Environmental Laboratory Accreditation Conference's (NELAC) Quality System requirements and provides implementation clarification and expectations for DoD environmental programs. It is designed to serve as a standard reference for DoD representatives from all components who design, implement, and oversee contracts with environmental testing laboratories.

Background

To be accredited under the National Environmental Laboratory Accreditation Program (NELAP), laboratories shall have a comprehensive quality system in place, the requirements for which are outlined in NELAP Chapter 5 (Quality Systems). Using NELAP Chapter 5 as its textual base, the *DoD Quality Systems Manual for Environmental Laboratories* is designed to replace common components of the following documents, previously issued by individual components of DoD:

- United States Navy – Section 3, *Installation Restoration Laboratory Quality Assurance Guide*, Interim Document, February 1996.
- Air Force Center for Environmental Excellence – *Quality Assurance Project Plan, Version 3.* March 1998.
- Army Corps of Engineers (USACE – HTRW) – Appendix I of EM200-1-3. ~~*Interim Chemical Data Quality Management (CDQM) Policy for USACE HTRW Projects.* 8 December 1998.~~

In combining the common components of these three documents, this ~~M~~manual allows laboratories to design quality systems to meet basic requirements for laboratory accreditation under NELAP, as well as the implementation needs of all DoD components. The document achieves this by ~~clarifying~~ summarizing and elaborating on DoD's expectations of the laboratory, with respect to the implementation of specific components of the NELAC Quality System.

Full implementation of this ~~M~~manual's requirements is expected within 2 years following release. This standardized document is only one of several efforts planned for implementation by DoD. As such, until such time as further standardization by DoD occurs, this document may be supplemented by component-specific requirements.

Project Specific Requirements

Requirements contained in this ~~M~~manual are superseded by **project-specific requirements or regulations**. The laboratory bears the responsibility for meeting all **State requirements**. Nothing in this document relieves any laboratory from complying with contract requirements or with **Federal, State, and/or local regulations**.

Results and Benefits

The side-by-side integration of NELAP requirements with clarifications by DoD regarding implementation creates several benefits for the laboratory, DoD, and the regulatory communities.

- **Standardization of Processes** – Because this manual provides laboratories with a comprehensive set of requirements that meet the needs of all DoD clients, as well as NELAP, the laboratory may use it to create a standardized quality system. Ultimately, this standardization will save laboratory resources by establishing one set of consistent requirements for all DoD environmental work. The standardized guidance will also serve to "level the playing field" for laboratories competing for DoD contracts, because the expectations will be identical across all DoD components. An audit that

satisfies the needs of one component will satisfy comparable needs of the other components as well. As such, this ~~M~~manual will facilitate the standardization of audits, which are consistent and transferable between components. The result will be saved resources for both the Government and private sector.

- **Deterrence of Improper, Unethical, or Illegal Actions** – Improper, unethical, or illegal activities by only a few laboratories have implications throughout the industry, with negative impacts on all laboratories. This ~~M~~manual addresses this issue, establishing a minimum threshold program for all laboratories to use to deter and detect improper, unethical, or illegal actions.

- **Specification of Compliance Requirements** – Because this ~~M~~manual applies to all laboratories performing environmental work for DoD, it represents the first policy guidance for laboratories involved in compliance testing.

- **Foundations for the Future** – A standardized approach to quality systems, shared by laboratories, NELAP, and DoD, paves the way for the standardization of other processes in the future. For example, this ~~M~~manual might serve as a platform for a standardized strategy for Performance Based Measurement System (PBMS) implementation. In addition, as noted above, DoD plans to supplement this document with other standardized tools, including standard report formats.

Audience

This ~~M~~manual is designed to meet the needs of the following audiences:

- Public (i.e., Government) and private laboratories, contracted with DoD either directly, or through a prime contractor or subcontractor;
- DoD implementing agency representatives, who will use this document to ensure consistency with NELAP when drafting contracts; and
- DoD oversight personnel and assessors, who will use this document to uniformly and consistently evaluate the laboratory's implementation of NELAP and DoD program requirements.

Document Format

Because the *DoD Quality Systems Manual* is designed to complement and implement NELAP Chapter 5 (Quality Systems), that document serves as the primary text for this implementation ~~M~~manual. The section numbering has been slightly changed from that of NELAP Chapter 5 as the ~~M~~manual is meant to be a stand-alone document. The number 5 has been eliminated from all section and subsection headings. However, second-level numbering has been retained to ensure maintenance of a parallel organization to the NELAC Quality Systems requirements. For instance, Section 5.4.2 in NELAP Chapter 5 (referencing Chapter 5 of the NELAC standards) is equivalent to Section 4.2 in this ~~M~~manual. DoD clarifications that elaborate on specific NELAP requirements are presented in gray text boxes~~,~~ ~~placed at~~ in the applicable section of the document. This allows laboratories preparing for NELAP accreditation to implement their quality systems in a way that fulfills the needs of DoD~~,~~ as well as NELAP. For ease of reference, each gray clarification box in the draft document is numbered. In addition, there are two sets of appendices to this DoD ~~M~~manual. The first set is the NELAC appendices, modified with DoD clarification boxes. The second set is DoD appendices. The DoD appendices will include specific focus on areas of standardization that will be implemented across all DoD components for laboratory services. ~~DoD clarifications that elaborate on specific NELAP requirements are presented in gray text boxes, placed at the applicable section of the document. This allows laboratories preparing for NELAP accreditation to implement their Quality Systems in a way that fulfills the needs of DoD, as well as NELAP. For ease of reference, each gray clarification box in the draft document is numbered.~~

LIST OF DoD IMPLEMENTATION CLARIFICATION BOXES

LIST OF DoD IMPLEMENTATION CLARIFICATION BOXES (Continued)

ACROYNM LIST

°C: Degrees Celsius
ANSI/ASQC: American National Standards Institute/American Society for Quality Control
ASTM: American Society for Testing and Materials
CAS: Chemical Abstract Service
CCV: Continuing calibration verification
CFR: Code of Federal Regulations
CLP: Contract Laboratory Program
COC: Chain of custody
CV: Coefficient of variation
DO: Dissolved oxygen
DOC: Demonstration of capability
DoD: Department of Defense
DQOs: Data quality objectives
EC: Exposure concentration
EPA: Environmental Protection Agency
g/L: Grams per liter
GC/MS: Gas chromatography/mass spectrometry
ICP-MS: Inductively coupled plasma-mass spectrometer
ICV: Initial calibration verification
ID: Identifier
ISO/IEC: International Standards Organization/International Electrotechnical Commission
LC50: Lethal concentration at 50%
LCS: Laboratory control sample
LQMP: Laboratory Quality Management Plan
MDL: Method detection limit
mg/kg: Milligrams per kilogram
MQO: Measurement quality objective
MS: Matrix spike
MSD: Matrix spike duplicate
~~**MSD:** Minimum significant difference~~
NELAC: National Environmental Laboratory Accreditation Conference
NELAP: National Environmental Laboratory Accreditation Program
NIST: National Institute of Standards and Technology
NOEC: No-observable-effects concentration
OSHA: Occupational Safety and Health Administration
PBMS: Performance Based Measurement System
PC: Personal computer
PCBs: Polychlorinated biphenyls
PT: Proficiency testing
QA: Quality assurance
QAD: Quality Assurance Division (EPA)
QAMS: Quality Assurance Management Section
QAPP: Quality Assurance Project Plan
QC: Quality control
RL: Reporting limit
RPD: Relative percent difference
RSD: Relative standard deviation
SD: Serial dilutions
SOP: Standard operating procedure
TAC: Test acceptability criteria
TSS: Total suspended solids
UV: Ultraviolet
VOC: Volatile organic compound
WET: Whole effluent toxicity
SMSD: Statistical minimum significant difference

QUALITY SYSTEMS

Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures, which shall be delineated in a Quality Manual and followed to ensure and document the quality of the analytical data. Laboratories seeking accreditation under the National Environmental Accreditation Program (NELAP) must assure implementation of all QA policies and the applicable QC procedures specified in this chapter. The QA policies, which establish essential QC procedures, are applicable to environmental laboratories regardless of size and complexity.

The intent of this Chapter is to provide sufficient detail concerning quality management requirements so that all accrediting authorities evaluate laboratories consistently and uniformly.

NELAC is committed to the use of Performance Based Measurement Systems (PBMS) in environmental testing and provides the foundation for PBMS implementation in these standards. While this standard may not currently satisfy all the anticipated needs of PBMS, NELAC will address future needs within the context of State statutory and regulatory requirements and the finalized EPA implementation plans for PBMS.

Chapter 5 is organized according to the structure of ISO/IEC Guide 25, 1990. Where deemed necessary, specific areas within this Chapter may contain more information than specified by ISO/IEC Guide 25.

All items identified in this chapter shall be available for on-site inspection or data audit.

1.0 SCOPE

a) This Standard sets out the general requirements that a laboratory has to successfully demonstrate to be recognized as competent to carry out specific environmental tests.

b) This Standard includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority granting the recognition (or approval).

   If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. (See the supplemental accreditation requirements in Section 1.9.2 of NELAC.)

c) This Standard is for use by environmental testing laboratories in the development and implementation of their quality systems. It shall be used by accreditation authorities, in assessing the competence of environmental laboratories.

Scope of DoD Document:

- These standards are applicable to any laboratory providing sample analysis to support environmental programs for DoD installations and facilities within the United States and its possessions.
- These standards are intended to apply to laboratories that produce definitive data, regardless of the methods being applied (i.e., technically defensible and legally admissible data).
- These standards may be supplemented by project-specific requirements, as agreed upon by the agency, regulators, laboratories, and other involved parties.
- The laboratory bears the responsibility for meeting all State requirements. Nothing in this document relieves any laboratory from complying with contract requirements or with Federal, State, and/or local regulations.

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2.0 REFERENCES

See Appendix A.

3.0 DEFINITIONS

The relevant definitions from ISO/IEC Guide 2, ISO 8402, ANSI/ASQC E-4, 1994, the EPA "Glossary of Quality Assurance Terms and Acronyms," and the *International vocabulary of basic and general terms in metrology (VIM*) are applicable, the most relevant being quoted in Appendix A, Glossary, of Chapter 1 of NELAC, together with further definitions applicable for the purposes of this Standard.

Definitions: For reference purposes, applicable terms from the NELAC Glossary are included as Appendix B in this DoD ~~M~~manual. Furthermore, additional terms not currently included in the NELAP Glossary are defined by DoD to aid the laboratory in implementing this standard appropriately. These terms are also in Appendix B.

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4.0 ORGANIZATION AND MANAGEMENT

4.1 Legal Definition of Laboratory

The laboratory shall be legally identifiable. It shall be organized and shall operate in such a way that its permanent, temporary and mobile facilities meet the requirements of this Standard**.**

4.2 Organization

The laboratory shall:

a) Have managerial staff with the authority and resources needed to discharge their duties;

b) Have processes to ensure that its personnel are free from any commercial, financial and other undue pressures which adversely affect the quality of their work;

c) Be organized in such a way that confidence in its independence of judgment and integrity is maintained at all times;

d) Specify and document the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests;

   Such documentation shall include:

   1) A clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured and

   2) Job descriptions for all positions.

e) Provide supervision by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test, and the assessment of the results.

   The ratio of supervisory to non-supervisory personnel shall be such as to ensure adequate supervision to ensure adherence to laboratory procedures and accepted techniques.

f) Have a technical director(s) (however named) who has overall responsibility for the technical operation of the environmental testing laboratory.

The technical director(s) shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented.

The technical director(s) shall meet the requirements specified in the Accreditation Process. (See NELAC Section 4.1.1.1.)

g) Have a quality assurance officer (however named) who has responsibility for the quality system and its implementation.

The quality assurance officer shall have direct access to the highest level of management at which decisions are taken on laboratory policy or resources, and to the technical director. Where staffing is limited, the quality assurance officer may also be the technical director or deputy technical director.

The quality assurance officer (and/or his/her designees) shall:

1) Serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;

2) Have functions independent from laboratory operations for which they have quality assurance oversight;

3) Be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

4) Have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system, as defined under NELAC;

5) Have a general knowledge of the analytical test methods for which data review is performed;

6) Arrange for or conduct internal audits as per 5.3 annually; and

7) Notify laboratory management of deficiencies in the quality system and monitor corrective action.

Quality Assurance – Duty of Quality Assurance Officer: The quality assurance officer shall also be responsible for ensuring continuous improvement at the laboratory through the use of control charts and other method performance indicators (for example, proficiency testing (PT) samples and internal and external audits).

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h) Nominate deputies in case of absence of the technical director(s) and/or quality assurance officer;

i) Have documented policy and procedures to ensure the protection of clients' confidential information and proprietary rights (this may not apply to in-house laboratories);

j) For purposes of qualifying for and maintaining accreditation, each laboratory shall participate in a proficiency test program as outlined in Chapter 2 of NELAC.

Technical Directors – Responsibility of Technical Directors: Lab management is responsible for following through with proficiency testing programs and for ensuring that corrective actions are implemented after testing and evaluating the effectiveness of the corrective actions.

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5.0 QUALITY SYSTEM – ESTABLISHMENT, AUDITS, ESSENTIAL QUALITY CONTROLS, AND DATA VERIFICATION

5.1 Establishment

The laboratory shall establish and maintain a quality system based on the required elements contained in this chapter and appropriate to the type, range and volume of environmental testing activities it undertakes.

a) The elements of this quality system shall be documented in the organization's quality manual.

b) The quality documentation shall be available for use by the laboratory personnel.

Quality System Documentation: This documentation includes the ~~Q~~quality ~~M~~manual, standard operating procedure (SOP) documents, and other appropriate reference documents and texts.

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c) The laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

d) The laboratory management shall ensure that these policies and objectives are documented in a quality manual and communicated to, understood and implemented by all laboratory personnel concerned.

e) The quality manual shall be maintained current under the responsibility of the quality assurance officer.

5.2 Quality Manual

The quality manual, and related quality documentation, shall state the laboratory's policies and operational procedures established in order to meet the requirements of this Standard.

The quality manual shall list on the title page: a document title; the laboratory's full name and address; the name, address (if different from above), and telephone number of individual(s) responsible for the laboratory; the name of the quality assurance officer (however named); the identification of all major organizational units which are to be covered by this quality manual and the effective date of the version.

Quality Manual Updating: The following list reflects topic areas that shall be included in the ~~Q~~quality ~~M~~manual. Additional details about each topic area are provided in the sections that follow. The ~~M~~manual shall be reviewed at least annually for accuracy and adequacy, and updated as appropriate. All such reviews shall be documented and available for inspection.

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The quality manual and related quality documentation shall also contain:

a) A quality policy statement, including objectives and commitments, by top management;

b) The organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;

Corporations – Laboratory Relationships with Corporations: This includes the laboratory's relationship(s) to corporate affiliations and networks.

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c) The relationship between management, technical operations, support services and the quality system;

d) Procedures to ensure that all records required under this Chapter are retained, as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;

Document Control – Distribution: Consistent with the definition of "Document Control" provided in NELAP Appendix B, this control system shall ensure that all analysts implementing the task(s) or procedure(s) described in that SOP shall be made individually aware that changes to an SOP have occurred. A copy of the updated SOP shall be available in close proximity to the work station (i.e., within the same work area).

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e) Job descriptions of key staff and reference to the job descriptions of other staff;

Personnel To Be Included in Quality Manual: At a minimum, the following managerial and supervisory staff (however named) shall be considered key staff, and their job descriptions shall be included in the ~~Q~~quality ~~M~~manual and other related documents:

(1) Executive Staff (for example, Chief Executive Officer, Chief Operating Officer, laboratory director, technical director);
(2) Technical directors/supervisors (for example, section supervisors for organics and inorganics);
(3) Quality assurance systems directors/supervisors (for example, QA officer, quality auditors); and
(4) Support systems directors/supervisors (for example, information systems supervisor, purchasing director, and project managers).

In addition, the ~~Q~~quality ~~M~~manual shall include job descriptions for key staff in each of these four areas, as appropriate to the laboratory.

If the size and organization of the laboratory precludes separate managers and/or supervisors in each of these key areas, the functions covered in the four areas shall be addressed in the job descriptions provided for the key staff.

The ~~Q~~quality ~~M~~manual shall describe the relationship of the key staff listed above to other technical and support staff. Any changes in key personnel for the laboratory must be documented to all laboratory users.

Technical staff includes those individuals who conduct the work of the laboratory (for example, sample receipt and documentation staff, the chemists who run the analytical equipment). Support staff administers the business practices of the laboratory, as well as information management and contractual systems. Quality assurance staff oversees the implementation of the quality system, and reports to the quality assurance officer or his/her designee.

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f) Identification of the laboratory's approved signatories; at a minimum, the title page of the quality manual must have the signed and dated concurrence (with appropriate titles) of all responsible parties including the QA officer(s), technical director(s), and the agent who is in charge of all laboratory activities, such as the laboratory director or laboratory manager;

g) The laboratory's procedures for achieving traceability of measurements;

Traceability of Measurements: Standards addressing this issue are included in Section 9.0 (Measurement Traceability and Calibration), Section 10.5 (Documentation and Labeling of Standards and Reagents), and Section 12.0 (Records).

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h) A list of all test methods under which the laboratory performs its accredited testing;

i) Mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

j) Reference to the calibration and/or verification test procedures used;

k) Procedures for handling submitted samples;

l) Reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;

m) Reference to procedures for calibration, verification and maintenance of equipment;

n) Reference to verification practices which may include interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

o) Procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

p) The laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

q) Procedures for dealing with complaints;

r) Procedures for protecting confidentiality (including national security concerns) and proprietary rights;

s) Procedures for audits and data review;

Audits – Quality Manual Specification: The ~~Q~~quality ~~M~~manual shall also specify which records are considered necessary to conduct an adequate review.

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t) Processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training;

u) Ethics policy statement developed by the laboratory and processes/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical, or illegal actions;

Personnel Training – Ethical: Additional descriptions related to this requirement are included in Section 6.2.

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v) Reference to procedures for reporting analytical results; and

w) A Table of Contents and applicable lists of references and glossaries, and appendices.

5.3 Audits

Audits – Section Summary: The following subsections of 5.3 refer to internal assessment tools to be used by the laboratory. Section 5.3.1 discusses systems audits and technical audits, both of which shall be conducted annually to evaluate whether the quality system is being implemented at the operational level of the laboratory. Section 5.3.2 addresses higher-level managerial reviews, designed to evaluate whether the quality system itself is effective. These can be done in conjunction with each other or separately, at the discretion of the laboratory. This section also addresses requirements for a program to detect and prevent improper, unethical, or illegal actions. Section 5.3.3 addresses the review of all auditing activities. Section 5.3.4 addresses continuous quality control practices, which shall be conducted by the laboratory on an ongoing basis.

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5.3.1 Internal Audits

The laboratory shall arrange for annual internal audits to verify that its operations continue to comply with the requirements of the laboratory's quality system. It is the responsibility of the quality assurance officer to plan and organize audits as required by a predetermined schedule and requested by management. Such audits shall be carried out by trained and qualified personnel who are, wherever resources permit, independent of the activity to be audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out. Where the audit findings cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose work was involved.

Audits – Internal: Internal audits shall include both technical audits and systems audits. They may be scheduled or unannounced. Technical audits verify compliance with method-specific requirements, as well as operations related to the test method (for example, sample preparation). (These operations include all actions related to data generation and the assurance of its quality.) Systems audits verify compliance with the laboratory's quality system, based on the NELAP Quality System, and documented in the ~~Q~~quality ~~M~~manual. Response to complaints, sample acceptance policies, and sample tracking methodologies are examples of procedures that would be reviewed as part of a systems audit. Data audits are considered a subset of technical audits.

An audit schedule shall be established such that all elements/areas of the laboratory are reviewed over the course of 1 year.

Personnel performing an internal audit shall complete the audit under the direction of the quality assurance officer, however named. To be considered "trained and qualified," the internal auditor shall be trained and qualified in conducting the type of audit under review.

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5.3.2 Managerial Review

The laboratory management shall conduct a review, at least annually, of its quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review shall take account of reports from managerial and supervisory personnel, the outcome of recent internal audits, assessments by external bodies, the results of inter-laboratory comparisons or proficiency tests, any changes in the volume and type of work undertaken, feedback from clients, corrective actions, and other relevant factors. The laboratory shall have a procedure for review by management and maintain records of review findings and actions.

Audits – Managerial Review: This is a separate review from the internal audit discussed in Section 5.3.1 and shall be completed by laboratory managerial personnel. As noted in clarification box 13, however, internal audits and managerial reviews may be conducted in conjunction with each other.

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5.3.3 Audit Review

All audit and review findings and any corrective actions that arise from them shall be documented. The laboratory management shall ensure that these actions are discharged within the agreed time frame as indicated in the quality manual and/or SOPs.

Audits – Timeframe of Audit Review: The timeframe for these actions shall be based on the magnitude of the problem and its impact on the defensibility and use of data.

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5.3.4 Performance Audits

In addition to periodic audits, the laboratory shall ensure the quality of results provided to clients by implementing checks to monitor the quality of the laboratory's analytical activities. Examples of such checks are:

a) Internal quality control procedures using statistical techniques (see Section 5.4 below);

b) Participation in proficiency testing or other interlaboratory comparisons (see Chapter 2 of NELAC);

c) Use of certified reference materials and/or in-house quality control using secondary reference materials as specified in Section 5.4;

d) Replicate testings using the same or different test methods;

e) Re-testing of retained samples;

f) Correlation of results for different but related analysis of a sample (for example, total phosphorus should be greater than or equal to orthophosphate).

Audits – Laboratory Checks of Performance Audits: This section requires the laboratory to continuously evaluate the quality of generated data by systematically and routinely implementing control checks that go beyond those required by the test methods. The results of these checks (examples of which are listed above) shall be routinely reviewed after they are performed to monitor and evaluate the quality and usability of data generated by the laboratory. Although a supplemental review of these checks shall be included as part of the annual internal audits, the laboratory shall also ensure that the results of these checks are reviewed (and corrective action taken) on a regular and timely basis following the actual completion of the check to remedy the problem, avoid its recurrence, and improve the quality system overall.

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5.3.5 Corrective Actions

a) In addition to providing acceptance criteria and specific protocols for corrective actions in the Method Standard Operating Procedures (see 10.1.1), the laboratory shall implement general procedures to be followed to determine when departures from documented policies, procedures and quality control have occurred. These procedures shall include but are not limited to the following:

    1) Identify the individual(s) responsible for assessing each QC data type;

2) Identify the individual(s) responsible for initiating and/or recommending corrective actions;

3) Define how the analyst shall treat a data set if the associated QC measurements are unacceptable;

4) Specify how out-of-control situations and subsequent corrective actions are to be documented; and

5) Specify procedures for management (including the QA officer) to review corrective action reports.

Audits – Corrective Action: Managers, including the QA officer, are also responsible for acting upon corrective action report reviews. Furthermore, managers are ultimately accountable for the follow-through, verification, and evaluation of these corrective actions. Further explanatory clarifications of DoD expectations are provided as follows:

Nonconformance. The laboratory shall have an established, documented policy and procedures to identify and control work and test results that do not or may not meet expected or specified requirements, or are nonconforming or suspected to be nonconforming. Policy and procedures shall ensure that:

- Responsibilities and authorities for the managing of nonconforming work/results are designated.
- Actions to be taken following identification of a nonconformance are defined and implemented, and include, but are not limited to, evaluating the significance of a nonconformance; halting work and investigating the contributors to the nonconformance (for example, equipment, personnel, methods); withholding reports and certificates, as necessary; informing clients of nonconformance resulting from their samples and the need to recall results of nonconforming work already released to them; implementing corrective action, as needed, and evaluating the results. (See corrective action requirements below.)

Corrective Action. The laboratory shall have established, documented policy and procedures for actions to be taken to eliminate the causes of a nonconformance and to prevent recurrence. The corrective action process shall identify and implement corrective actions likely to eliminate the root cause of nonconformance(s). Laboratory policies and procedures shall ensure that:

- Responsibilities and authorities for instituting corrective action are designated.
- Possible causes of the nonconformance(s) are investigated.
- Root cause analysis is performed.
- Changes resulting from corrective action are recorded and retrievable.
- Corrective action(s) are monitored.
- Preventive action is taken to prevent recurrence.

Monitoring of Corrective Actions. After implementation of corrective action(s), the laboratory shall monitor their effects to determine if action(s) taken are effective in overcoming the nonconformance identified (i.e., the root cause has been eliminated and its recurrence prevented). Historical corrective action reports should be periodically reviewed to identify long-term trends or recurring problems.

Preventive Action. All operations shall be systematically and thoroughly reviewed at regular intervals to:

- Obtain input on the laboratory's operations;
- Determine what considerations need to be given to input (from reviews); and
- Determine how corrective action(s), if necessary, shall be carried out.

Reference: American Society for Quality Control. 1991. *Q2 – Quality Management and Quality System Elements for Laboratories – Guidelines.* **18**

b) To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data are to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s).

Data Qualifiers: Some of the standard data qualifiers, to be used by laboratories only, are listed below. Additional data qualifiers may be used by data validators when evaluating data usability (for example, an "R" flag for rejected data).

U – Undetected at the method detection limit: The associated ~~number~~ data value is the ~~reporting~~ method detection limit, adjusted by any dilution factor used in the analysis.
J – Estimated: The analyte was positively identified; the quantitation is an estimation (for example, matrix interference, below standard, outside the calibration range).
B – Blank contamination: Analyte detected above the reporting limit in an associated blank.
N – Nontarget analyte: Analyte is a tentatively identified compound (using mass spectroscopy).
Q – One or more quality control criteria (for example, LCS recovery, surrogate spike recovery, etc.) failed. Data usability should be carefully assessed by an individual experienced in data review who represents the data user or their agent. Assessment by DoD may result in rejection of data and potential contractual nonpayment based on unacceptable performance.

When other flags are required contractually, these shall be used.

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5.4 Essential Quality Control Procedures

These general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e., chemical, whole effluent toxicity, microbiological, radiological, air) and are further described in Appendix D. The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have detailed written protocols in place to monitor the following quality controls:

Quality Control Actions: Quality control actions should be both batch-specific and time-based (i.e., those required to be conducted at specific time periods, such as for tunes and method detection limits [MDLs]). Batch-specific quality control actions include sample-specific quality control actions such as surrogate spikes.

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1) Positive and negative controls to monitor tests such as blanks, spikes, reference toxicants;

2) Tests to define the variability and/or repeatability of the laboratory results such as replicates;

3) Measures to assure the accuracy of the test method including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

4) Measures to evaluate test method capability, such as detection limits and quantitation limits or range of applicability such as linearity;

5) Selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses;

6) Selection and use of reagents and standards of appropriate quality;

7) Measures to assure the selectivity of the test for its intended purpose; and

8) Measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the test method, such as temperature, humidity, light, or specific instrument conditions.

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance criteria shall be used to determine the usability of the data. (See Appendix D.)

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See 11.2, Sample Acceptance Policy.)

d) The quality control protocols specified by the laboratory's method manual (10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D, or mandated methods or regulations (whichever are more stringent) are incorporated into their method manuals. When it is not apparent which is more stringent the QC in the mandated method or regulations is to be followed.

The essential quality control measures for testing are found in Appendix D.

6.0 PERSONNEL

6.1 General Requirements for Laboratory Staff

The laboratory shall have sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned functions.

All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, test methods, quality assurance/quality control procedures and records management.

Technical Directors – Qualifications: Required qualifications for the technical director(s) are addressed further below. DoD stresses that a director or designee meeting the qualifications below shall be present in each area of analytical service. Laboratory management, as addressed in Section 6.2, is defined as designees (for example, laboratory manager, technical director, supervisors, and quality assurance officers, however named) having oversight authority and responsibility for laboratory output.

The following requirements are direct excerpts from **NELAP Chapter 4 (Accreditation Process), Revision 13 – June 29, 2000**.

4.1.1 Personnel Qualifications

Persons who do not meet the education credential requirements but possess the requisite experience of Section 4.1.1.1 of the NELAC standards and are the technical director(s) on the date that the laboratory becomes subject to these NELAC Standards and obtains accreditation shall qualify as technical director(s) for the field(s) of testing of that laboratory or any other NELAC-accredited laboratory.

4.1.1.1 Definition, Technical Director(s)

The technical director(s) means a full-time member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of testing and reporting of results. The title of such person may include but is not limited to laboratory director,

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technical director, laboratory supervisor or laboratory manager. A laboratory may appoint one or more technical directors for the testing for which they are seeking accreditation. His/her name must appear in the national database. This person's duties shall include, but not be limited to, monitoring standards of performance in quality control and quality assurance; monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data. An individual shall not be the technical director(s) of more than one accredited environmental laboratory without authorization from the primary accrediting authority. Circumstances to be considered in the decision to grant such authorization shall include, but not be limited to, the extent to which operating hours of the laboratories to be directed overlap, adequacy of supervision in each laboratory, and the availability of environmental laboratory services in the area served. The technical director(s) who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full-time staff member meeting the qualifications of the technical director(s) to temporarily perform this function. If this absence exceeds 65 consecutive calendar days, the primary accrediting authority shall be notified in writing.

Qualification of the technical director(s):

a) Any technical director of an accredited environmental laboratory engaged in chemical analysis shall be a person with a bachelor's degree in the chemical, environmental, biological sciences, physical sciences, or engineering, with at least 24 college semester credit hours in chemistry and at least two years of experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

b) Any technical director of an accredited environmental laboratory limited to inorganic chemical analysis, other than metals analysis, shall be a person with at least an earned associate's degree in the chemical, physical, or environmental sciences, or two years of equivalent and successful college education, with a minimum of 16 college semester credit hours in chemistry. In addition, such a person shall have at least two years of experience performing such analysis.

c) The technical director of an accredited environmental laboratory engaged in microbiological or biological analysis shall be a person with a bachelor's degree in microbiology, biology, chemistry, environmental sciences, physical sciences, or engineering with a minimum of 16 college semester credit hours in general microbiology and biology and at least two years of experience in the environmental analysis of representative analytes for which the laboratory seeks or maintains accreditation. A master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

   A person with an associate's degree in an appropriate field of the sciences or applied sciences, with a minimum of four college semester credit hours in general microbiology, may be the technical director(s) of a laboratory engaged in microbiological analysis limited to fecal coliform, total coliform, and standard plate count. Two years of equivalent and successful college education, including the microbiology requirement, may be substituted for the associate's degree. In addition, each person shall have one year of experience in environmental analysis.

d) Any technical director of an accredited environmental laboratory engaged in radiological analysis shall be a person with a bachelor's degree in chemistry, physics, or engineering with 24 college semester credit hours of chemistry with two or more years of experience in the radiological analysis of environmental samples. A master's or doctoral degree in one of the above disciplines may be substituted for one year experience.

e) Any technical director(s) of an accredited environmental laboratory engaged in microscopic examination of asbestos and/or airborne fibers shall meet the following requirements:

   i) For procedures requiring the use of a transmission electron microscope, a bachelor's degree, successful completion of courses in the use of the instrument, and one year of experience, under supervision, in the use of the instrument. Such experience shall include the identification of minerals.

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ii) For procedures requiring the use of a polarized light microscope, an associate's degree or two years of college study, successful completion of formal coursework in polarized light microscopy, and one year of experience, under supervision, in the use of the instrument. Such experience shall include the identification of minerals.

iii) For procedures requiring the use of a phase contrast microscope, as in the determination of airborne fibers, an associate's degree or two years of college study, documentation of successful completion of formal coursework in phase contrast microscopy, and one year of experience, under supervision, in the use of the instrument.

f) Any technical director of an accredited environmental laboratory engaged in the examination of radon in air shall have at least an associate's degree or two years of college and one year of experience in radiation measurements, including at least one year of experience in the measurement of radon and/or radon progeny.

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6.2 Laboratory Management Responsibilities

In addition to Section 4.2.d, the laboratory management shall be responsible for:

a) Defining the minimal level of qualification, experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills such as using a balance, colony counting, aseptic or quantitative techniques, shall be considered.

b) Ensuring that all technical laboratory staff have demonstrated capability in the activities for which they are responsible. Such demonstration shall be documented (See Appendix C).

Note: In laboratories with specialized "work cells" (a well defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

Work Cell – Definition of Work Cell: Additional guidance on this issue is provided in Section 10.2.1.f and g. A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation, extraction, and analysis. To ensure that the entire preparation, extraction, and analysis process is completed by a collection of capable individuals, the laboratory shall ensure that **each member** of the work cell (including a new member of an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a "team," the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance. A work cell may NOT be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

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c) Ensuring that the training of each member of the technical staff is kept up-to-date (on-going) by the following:

1) Evidence must be on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation, which relates to his/her job responsibilities.

2) Training courses or workshops on specific equipment, analytical techniques, or laboratory procedures shall all be documented.

3) Training courses in ethical and legal responsibilities include the potential punishments and penalties for improper, unethical or illegal actions. Evidence must also be on file which demonstrates that

each employee has read, acknowledged, and understood their personal ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions.

Personnel Training – Ongoing: Additional descriptions related to this requirement are included in Section 6.2.h

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4) Analyst training shall be considered up to date if an employee training file contains a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or standard operating procedure as defined by the laboratory document control system, 5.2.d) and documentation of continued proficiency by at least one of the following once per year:

   i. Acceptable performance of a blind sample (single blind to the analyst);

   ii. Another demonstration of capability;

   iii. Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for Methods 524.2, 624, or 5035/8260) would only require documentation for one of the test methods;

   iv. At least four consecutive laboratory control samples with acceptable levels of precision and accuracy;

   v. If i-iv cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.

d) Documenting all analytical and operational activities of the laboratory;

e) Supervising all personnel employed by the laboratory;

f) Ensuring that all sample acceptance criteria (Section 11.0) are verified and that samples are logged into the sample tracking system and properly labeled and stored.

g) Documenting the quality of all data reported by the laboratory.

h) Developing a proactive program for the prevention and detection of improper, unethical, or illegal actions. Components of this program could include: internal proficiency testing (single and double blind); post-analysis electronic and magnetic tape audits; effective reward program to improve employee vigilance and co-monitoring; and separate SOPs identifying appropriate and inappropriate laboratory and instrument manipulation practices.

A Program to Detect and Prevent Improper, Unethical, or Illegal Actions: In order to perform work for DoD under this ~~M~~manual, the laboratory shall have a documented program to prevent improper, unethical, or illegal actions. To facilitate the implementation of this required program, DoD has compiled the following text to (1) clearly define improper, unethical, or illegal actions; (2) outline elements of prevention and detection programs for improper, unethical, or illegal actions; and (3) identify examples of inappropriate (i.e., potentially fraudulent) laboratory practices. Data shall be produced according to the project-specific requirements as specified in the final approved project documents, such as the approved QAPP. The laboratory shall be aware of these requirements and be able to show that these requirements were followed.

Improper Actions. Improper actions are defined as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional. Unethical or illegal actions are defined as the deliberate falsification of analytical or quality assurance results, where failed method or contractual requirements are made to appear acceptable. Prevention of laboratory improper, unethical, or illegal actions begins with a zero-tolerance philosophy established by management. Improper, unethical, or illegal actions are detected through the implementation of oversight protocols.

Prevention and Detection Program for Improper, Unethical, or Illegal Actions. Laboratory management shall implement a variety of proactive measures to promote prevention and detection of improper, unethical, or illegal activities. The following components constitute the baseline and minimum requirements for an improper, unethical, or illegal actions prevention program and shall be included as part of the laboratory's comprehensive quality program:

- An ethics policy that is read and signed by all personnel;
- Initial and annual ethics training;
- Internal audits, as described elsewhere in Section 5.3;
- Inclusion of anti-fraud language in subcontracts;
- Analyst notation and sign-off on manual integration changes to data (See also DoD clarification boxes 30 and 38);
- Active use of electronic audit functions when they are available in the instrument software (see also Section 12.0); and
- A "no-fault" policy that encourages laboratory personnel to come forward and report fraudulent activities.

A proactive, "beyond the basics" approach to the prevention of improper, unethical, or illegal actions is a necessary part of laboratory management. As such, in addition to the mandatory requirements above, the laboratory shall institute other actions to deter and detect improper, unethical, or illegal actions, as required by NELAC Section 6.2.4(h) (i.e., designate an ombudsman (data integrity officer) to whom laboratory personnel can report improper, unethical, or illegal practices, or provide routine communication of training, lectures, and changes in policy intended to reduce improper, unethical, or illegal actions).

Examples of Improper, Unethical, or Illegal Practices. Documentation that clearly shows how all analytical values were obtained shall be maintained by the laboratory and supplied to the data user when necessary. To avoid miscommunication, a laboratory shall clearly document all errors, mistakes, and basis for manual integrations within the case narrative. Notification should also be made to the appropriate people such that appropriate corrective actions can be initiated. Gross deviations from specified procedures should be investigated for potential improper, unethical, or illegal actions, and findings of fraud should be prosecuted to the fullest extent of the law. Examples of improper, unethical, or illegal practices are identified below:

- Improper use of manual integrations to meet calibration or method QC criteria (for example, peak shaving or peak enhancement are considered improper, unethical, or illegal actions if performed solely to meet QC requirements);

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- Intentional misrepresentation of the date or time of analysis (for example, intentionally resetting a computer system's or instrument's date and/or time to make it appear that a time/date requirement was met);
- Falsification of results to meet method requirements;
- Reporting of results without analyses to support (i.e., dry-labbing);
- Selective exclusion of data to meet QC criteria (for example, initial calibration points dropped without technical or statistical justification);
- Misrepresentation of laboratory performance by presenting calibration data or QC limits within data reports that are not linked to the data set reported, or QC control limits presented within LQMP that are not indicative of historical laboratory performance or used for batch control;
- Notation of matrix inference as basis for exceeding acceptance limits (typically without implementing corrective actions) in interference-free matrices (for example, method blanks or laboratory control samples);
- Unwarranted manipulation of computer software (for example, improper background subtraction to meet ion abundance criteria for GC/MS tuning, chromatographic baseline manipulations);
- Improper alteration of analytical conditions (for example, modifying EM voltage, changing GC temperature program to shorter analytical run time) from standard analysis to sample analysis;
- Misrepresentation of QC samples (for example, adding surrogates after sample extraction, omitting sample preparation steps for QC samples, over- or underspiking); and
- Reporting of results from the analysis of one sample for those of another.

References:
California Military Environmental Coordination Committee (EPA, CAL EPA, and DoD). March 1997. "Best Practices for the Detection and Deterrence of Laboratory Fraud."
Army Corps of Engineers (USACE – HTRW) – *Interim Chemical Data Quality Management (CDQM) Policy for USACE HTRW Projects.* 8 December 1998.

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6.3 Records

Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory (see 6.2.c), including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in 10.2.1 for chemical testing.

7.0 PHYSICAL FACILITIES – ACCOMMODATION AND ENVIRONMENT

7.1 Environment

a) Laboratory accommodation, test areas, energy sources, lighting, heating and ventilation shall be such as to facilitate proper performance of tests.

Environment–Cooling: Laboratory accommodation, test areas, energy sources, lighting, heating, **cooling**, and ventilation shall be such as to facilitate proper performance tests.

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b) The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement. Particular care shall be taken when such activities are undertaken at sites other than the permanent laboratory premises.

c) The laboratory shall provide for the effective monitoring, control and recording of environmental conditions as appropriate. Such environmental conditions may include biological sterility, dust, electromagnetic interference, humidity, mains voltage, temperature, and sound and vibration levels.

d) In instances where monitoring or control of any of the above mentioned items are specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.

NOTE: It is the laboratory's responsibility to comply with the relevant health and safety requirements. This aspect, however, is outside the scope of this Standard.

7.2 Work Areas

a) There shall be effective separation between neighboring areas when the activities therein are incompatible including culture handling or incubation areas and volatile organic chemicals handling areas.

b) Access to and use of all areas affecting the quality of these activities shall be defined and controlled.

c) Adequate measures shall be taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.

d) Work spaces must be available to ensure an unencumbered work area. Work areas include:

1) Access and entryways to the laboratory;

2) Sample receipt area(s);

3) Sample storage area(s);

4) Chemical and waste storage area(s); and

5) Data handling and storage area(s).

8.0 EQUIPMENT AND REFERENCE MATERIALS

Equipment Standards: Equipment shall be capable of achieving the accuracy, precision, sensitivity, and selectivity required for the intended use of the generated data. The laboratory shall implement documented procedures to ensure that setup, maintenance, and adjustments to instrument operating parameters are documented, and that adjustments to instruments do not exceed the limits specified in the approved SOPs.

The use of outside support services and supplies is further addressed in Section 15.0.

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a) The laboratory shall be furnished with all items of equipment (including reference materials) required for the correct performance of tests for which accreditation is sought. In those cases where the laboratory needs to use equipment outside its permanent control it shall ensure that the relevant requirements of this Standard are met.

b) All equipment shall be properly maintained, inspected, and cleaned. Maintenance procedures shall be documented.

c) Any item of the equipment that has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by verification or otherwise to be defective, shall be taken out of service, clearly identified and wherever possible stored at a specified place until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations or tests.

d) Each item of equipment, including reference materials, shall, when appropriate, be labeled, marked, or otherwise identified to indicate its calibration status.

e) Records shall be maintained of each major item of equipment and all reference materials significant to the tests performed. These records shall include documentation on all routine and non-routine maintenance activities and reference material verifications.

The records shall include:

1) The name of the item of equipment;

2) The manufacturer's name, type identification, and serial number or other unique identification;

3) Date received and date placed in service (if available);

4) Current location, where appropriate;

5) If available, condition when received (e.g., new, used, reconditioned);

6) Copy of the manufacturer's instructions, where available;

7) Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;

8) Details of maintenance carried out to date and planned for the future; and

9) History of any damage, malfunction, modification or repair.

9.0 MEASUREMENT TRACEABILITY AND CALIBRATION

9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests shall be calibrated and/or verified before being put into service and on a continuing basis. The laboratory shall have an established program for the calibration and verification of its measuring and test equipment. This includes balances, thermometers and control standards.

9.2 Traceability of Calibration

a) The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that measurements made by the laboratory are traceable to national standards of measurement.

b) Calibration certificates shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory shall maintain records of all such certifications.

c) Where traceability to national standards of measurement is not applicable, the laboratory shall provide satisfactory evidence of correlation of results, for example, by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.

9.3 Reference Standards

a) Reference standards of measurement held by the laboratory (such as Class S or equivalent weights or traceable thermometers) shall be used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards have not been invalidated. Reference standards of measurement shall be calibrated by a body that can provide traceability. Where possible, this traceability shall be to a national standard of measurement.

b) There shall be a program of calibration and verification for reference standards.

c) Where relevant, reference standards and measuring and testing equipment shall be subjected to in-service checks between calibrations and verifications. Reference materials shall be traceable. Where possible, traceability shall be to national or international standards of measurement, or to national or international standard reference materials.

9.4 Calibration

Calibration requirements are divided into two parts: (1) requirements for analytical support equipment, and (2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

9.4.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume.

a) All support equipment shall be maintained in proper working order. The records of all repair and maintenance activities, including service calls, shall be kept.

b) All support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application for which this equipment is used or:

   1) The equipment shall be removed from service until repaired; or

   2) The laboratory shall maintain records of established correction factors to correct all measurements.

c) Raw data records shall be retained to document equipment performance.

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST traceable references where available. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

e) Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be checked for accuracy on at least a quarterly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory.

Volumetric Pipettes – Frequency of Accuracy Checks: As listed in the table associated with DoD clarification box 29, volumetric pipettes, both fixed and variable, shall be checked for accuracy on at least a **monthly** use basis.

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f) For chemical tests the temperature, cycle time and pressure of each run of autoclaves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

Autoclaves: The use of autoclaves during chemical tests is not typical, but is an analytical option for limited methods (for example, mercury soil digestion). The typical use would be for sterilization purposes as described in item g below.

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g) For biological tests that employ autoclave sterilization, see Section D.3.8.

Calibration – Calibration and Measurement Guidance: The following table provides specific guidance with respect to the calibration and performance measurements associated with specific types of analytical support equipment. The criteria presented that go beyond those established by the American Society for Testing and Methods (ASTM) standards are currently in use by DoD components. They are presented here in consolidated form and will be formally adopted across DoD as a standardized requirement. ASTM standards presented here are based on the latest edition available as of this ~~M~~manual's publication date. As new editions are released, the latest revision of each ASTM standard reference shall be followed, unless State or project requirements differ.

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Analytical Support Equipment Assessment	Frequency of Check	Acceptance Criteria	Calibration Check Procedures and Performance Criteria References (latest edition)
Balance calibration check	Daily or before use with two weights that bracket target weight(s) AND Annual calibration by certified technician	1% performance criterion to top-loading balances, and 0.1% to analytical balances. (Expanded criterion from 0.1 to 1% for top-loaders; no standard existed for this balance type.)	ASTM E 898, Standard Practice for the Evaluation of Single-Pan Mechanical Balances, E 319, Standard Practice for the Evaluation of Single-Pan Mechanical Balances, and D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid
Refrigerator/freezer temperature monitoring	Daily	Refrigerators: 4 ± 2 °C, Freezers: -10 to –20 °C (This ASTM standard does not address freezers, but SW-846 has noted this freezer range in some methods.)	ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid
Thermometer calibration check	Mercury – annually Electronic – quarterly at two temperatures that bracket target temperature(s) against a NIST-traceable thermometer	Appropriate correction factors applied.	ASTM Methods E 77, Standard Test Method for Inspection and Verification of Thermometers, and D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid

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Analytical Support Equipment Assessment	Frequency of Check	Acceptance Criteria	Calibration Check Procedures and Performance Criteria References (latest edition)
Volumetric pipettes (fixed or variable) (for example, Eppendorf)	Monthly	3% of known or true value. (Standard criteria for Class B transfer pipettes were used. Tolerance varied depending on volume delivered, with widest % associated with smaller volume pipettes – 2.4% tolerance applied to 0.5 mL pipette, so expanded to 3% for consistency.)	ASTM E 542, Standard Practice for Calibration of Volumetric Apparatus, and E 969, Standard Specification for Volumetric (Transfer) Pipettes
Nonvolumetric glassware/labware verification (Requirement applicable only when used for measuring initial sample and final extract/digestate volumes)	By lot at the time of purchase	3% of known or true value. (Standard tolerance does not exist. Class B volumetric flasks criteria vary between 0.8 to 0.05% for 5 mL to 2,000 mL, respectively – set at 3% to maintain consistency with pipette tolerance designation.)	ASTM E 542, Standard Practice for Calibration of Volumetric Ware
Drying ovens	Before and after use	Compliance with method-specific requirements.	ASTM D 5522, Standard Specification for Minimum Requirements for Laboratories Engaged in Chemical Analysis of Soil, Rock, and Contained Fluid 29

9.4.2 Instrument Calibration

This standard specifies the essential elements that will define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data must be of known quality and be appropriate for a given regulation or decision. This standard does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not apparent which standard is more stringent, then the requirements of the regulation or mandated test method are to be followed.

Note: In the following sections, initial instrument calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration.

Calibration – Instrument: The DoD implementation clarification boxes included in Section 9.4.2 will specify whether they only apply when method-specific guidance does not exist (for example, when PBMS is being used) or whether they apply to all methods.

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9.4.2.1 Initial Instrument Calibrations

The following items are essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures including calculations, integrations, acceptance criteria and associated statistics must be included or referenced in the test method SOP. When initial instrument calibration procedures are referenced in the test method, then the referenced material must be retained by the laboratory and be available for review.

b) Sufficient raw data records must be retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, analyst's initials or signature; concentration and response, calibration curve or response factor; or unique equation or coefficient used to reduce instrument responses to concentration.

Calibration (Initial) – Raw Data Records: ~~Raw data records shall also include the analyst's name.~~

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of the manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and signature/initials of person performing manual operation.

Applicable to all methods.

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c) Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing instrument calibration verification.

d) All initial instrument calibrations must be verified with a standard obtained from a second manufacturer or lot. Traceability shall be to a national standard, when available.

Calibration – Second Source Standards: ~~Second source standards~~ Initial instrument calibrations shall be ~~obtained~~ verified with a standard obtained from a different **manufacturer**, not from a different lot.~~than the original standard, unless one is not available.~~ The use of a standard from a second lot is acceptable when only one manufacturer of the calibration standard exists. Note: "~~M~~manufacturer" refers to the producer of the standard, not the vendor.

The requirement for a second source standard for the initial calibration verification is waived if a second source standard is used for the continuing calibration verification. ~~See DoD clarification box 37.~~ Deviations from this requirement require project-specific approval from appropriate DoD personnel (for example, project manager, quality assurance officer).

The date of preparation of each standard shall be considered when evaluating its suitability for use. This consideration shall include an assessment of the stability of the standard solution, as well as its degradation rate.

The concentration of the second source standard shall be at or near the middle of the calibration range. Criteria for the acceptance of second source verification standard results shall be established. Values chosen should be at least as stringent as those established for the continuing instrument calibration verification. The initial calibration verification shall be successfully completed prior to running any samples.

Applicable only when method-specific guidance does not exist.

321~~1~~

e) Criteria for the acceptance of an initial instrument calibration must be established, e.g., correlation coefficient or relative percent difference. The criteria used must be appropriate to the calibration technique employed.

Calibration – Initial Calibration Points: Criteria for the acceptance of an initial instrument calibration must be established (for example, correlation coefficient, **relative standard deviation**, etc.).

Exclusion of initial calibration points without technical justification is not allowed.

For example, in establishing an initial calibration curve, the calibration points used shall be a contiguous subset of the original set. In addition, the minimum linearity of the curve shall be determined either by a linear regression correlation coefficient greater than or equal to 0.995 or by a maximum mean percent relative standard deviation (%RSD) of 20% (with no individual analyte greater than 30%).

Deviations from the above, including for problem compounds, are permitted with the approval of DoD personnel (for example, project manager, quality assurance officer). See DoD clarification box 3~~6~~5 for guidance on the number of points required for a calibration curve.

Applicable only when method-specific guidance does not exist.

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f) Results of samples not bracketed by initial calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags or explained in the case narrative. The lowest calibration standard must be above the detection limit.

Calibration – Quantitative Values in a Calibration Curve:

The range of the accepted initial calibration curve reflects the quantitation range of the samples (i.e., only those sample results with concentrations contained within the range of the calibration curve are considered to be quantitative). Any data reported outside the calibration range shall be qualified as an estimated value (i.e., by a data qualifier "flag") and explained in the case narrative.

When sample concentrations **exceed** the upper limit of the calibration curve (i.e., upper quantitation limit), samples shall be diluted and reanalyzed (if possible) to bring them within the calibration curve. When sample concentrations **exceed** the upper limit of the calibration curve or **fall below** the lower limit of the calibration curve (i.e., below the lower quantitation limit), ~~then either the method shall be modified (for example, initial calibration rerun, thereby re-establishing the potential range of quantitative values), or In either instance,~~ the resulting data shall be qualified as having estimated values and shall be flagged with a J-flag.

The laboratory's **reporting limit** shall lie within the calibration range, at or above the lower quantitation limit. If the client **requires** a reporting limit that lies **below** the lower limit of the calibration curve (i.e., below the quantitation limit), then method modification is required. For methods that require only one standard (i.e., lower limit of curve is the origin), the reporting limit shall be no lower than a low-level check standard, designed to verify the integrity of the curve at the lower limits.

See also DoD clarification box D-1~~9~~20, which addresses detection limits, as well as definitions for quantitation limit and reporting limit.

Applicable only when method-specific guidance does not exist.

34~~3~~

g) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed. Data associated with an unacceptable initial instrument calibration shall not be reported.

h) Calibration standards must include concentrations at or below the regulatory limit/decision level, if these limits/levels are known by the laboratory, unless these concentrations are below the laboratory's demonstrated detection limits (See D.1.4 Detection Limits).

Calibration Standards – Laboratory Involvement: DoD recognizes that achievability of these limits/levels by the required method is a key variable. To avoid conflicts related to this issue, DoD expects laboratory involvement (government or private) during the planning phase of the project (QAPP preparation) to ensure proper selection of methods and instrumentation. If the proposed laboratory for the project work is unavailable for this consultation (for example, not yet selected), a Government laboratory may be consulted to establish these parameters. This early involvement of a laboratory is integral in ensuring efficient planning and implementation of the project.

Applicable to all methods.

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i) If a reference or mandated method does not specify the number of calibration standards, the minimum number is two, not including blanks or a zero standard. The laboratory must have a standard operating procedure for determining the number of points for establishing the initial instrument calibration.

Calibration – Initial Calibration: In completing work for DoD, the initial calibration range shall consist of a minimum of 5 contiguous calibration points for organics and a minimum of 3 contiguous calibration points for inorganics. All reported target analytes and surrogates shall be included in the initial calibration. For multicomponent analytes, such as PCBs, toxaphene, and dioxins/furans, a separate initial calibration may be required. See DoD clarification box 3~~2~~ in Section 9.4.2.1.e for additional implementation requirements pertaining to this subject.

Applicable when method-specific guidance does not exist.

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9.4.2.2 Continuing Instrument Calibration Verification

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

Calibration – Continuing Instrument Calibration Verification: The validity of the initial calibration shall be verified prior to sample analyses by an **acceptable** continuing instrument calibration verification with each analytical batch. As long as the continuing calibration verification (CCV) is acceptable, a new initial instrument calibration is not necessary.

Applicable when method-specific guidance does not exist.

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a) The details of the continuing instrument calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.

b) A continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch. The concentrations of the calibration verification shall be varied within the established calibration range. If an internal standard is used, only one continuing instrument calibration verification must be analyzed per analytical batch.

Calibration – Continuing Calibration Verification Frequency: ~~At least one of the CCV standards shall fall below the middle of the calibration range.~~ At a minimum, additional periodic CCVs shall be run whenever required by the applicable method. When the methods specify that CCVs shall be run at specific sample intervals (for example, every 10 samples), the count of these samples shall be of field samples only. However, QC samples must be run with their associated batch. The grouping of QC samples from a variety of batches is not an acceptable practice. If the method does not specify an interval for periodic CCVs, at a minimum, every preparatory batch should be bracketed (i.e., at least every 20 field samples). More frequent CCVs are recommended for more difficult matrices.

Applicable to all methods.

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c) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor, or unique equations or coefficients used to convert instrument responses into concentrations. Continuing calibration verification records must explicitly connect the continuing verification data to the initial instrument calibration.

Calibration (Continuing) – Raw Data Records: Raw data records shall also include the analyst's name.

When manual integrations are performed, raw data records shall include a complete audit trail for those manipulations, raw data output showing the results of the manual integration (i.e., chromatograms of manually integrated peaks), and notation of rationale, date, and signature/initials of person performing manual operation.

Applicable to all methods.

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d) Criteria for the acceptance of a continuing instrument calibration verification must be established, e.g., relative percent difference.

Calibration – CCV Criteria:

- At least one of the CCV standards shall fall below the middle of the calibration range.
- The source of the standard(s) for analysis can be the standard(s) used for the initial calibration.
- All reportable target analytes shall be included in the CCV. Where multicomponent, multi-analyte tests are being performed, a single multicomponent continuing calibration is acceptable.
- The baseline for comparison for the CCV is the initial calibration (and the original standards). Specific criteria for evaluation of success or failure of the CCV may include percent difference/drift from the RSD established for the initial calibration, minimum response factor checks, and confirmation that the retention time is within an acceptable window. For DoD, the percent drift/percent difference of the CCV standard shall be less than 15% of the initial calibration for organic methods and less than 10% of the initial calibration for inorganic methods, or shall be equivalent to the percent drift the standard method would have allowed. If the mean value for all target analytes is used, no percent drift for an individual analyte shall exceed 25%.

Applicable when method-specific guidance does not exist.

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e) If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory shall demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new instrument calibration must be performed. If the laboratory has not

demonstrated acceptable performance, sample analyses shall not occur until a new initial calibration curve is established and verified.

Calibration – Reporting Data from Noncompliant CCV: If initial corrective action attempts fail and the CCV results are still outside established acceptance criteria, and the laboratory chooses to demonstrate the success of routine corrective action through the use of two consecutive CCVs, then the concentrations of the two CCVs must be at two different levels within the original calibration curve. As stated in DoD clarification box 4037, at least one of the CCV standards shall fall below the middle of the initial calibration range.

Applicable to all methods.

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However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

i. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

ii. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

Calibration – Q Flag Reporting for Noncompliant CCV: Project-specific permission from appropriate DoD personnel is required to report data generated from the initial run with the noncompliant CCV. If this permission is granted, and these data are reported, they shall be qualified through the use of a "Q" flag and explained in the case narrative.

Applicable to all methods.

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10.0 TEST METHODS AND STANDARD OPERATING PROCEDURES

10.1 Methods Documentation

a) The laboratory shall have documented instructions on the use and operation of all relevant equipment, on the handling and preparation of samples and for calibration and/or testing, where the absence of such instructions could jeopardize the calibrations or tests.

b) All instructions, standards, manuals, and reference data relevant to the work of the laboratory shall be maintained up-to-date and be readily available to the staff.

Test Method/SOP Updating: All methods documentation (for example, instructions, standards, manuals, SOPs, etc.) shall be reviewed for accuracy and adequacy at least annually, or whenever procedural method changes occur, and updated as appropriate.

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10.1.1 Standard Operating Procedures (SOPs)

Laboratories shall maintain standard operating procedures that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

a) These documents, for example, may be equipment manuals provided by the manufacturer or internally written documents.

b) The test methods may be copies of published methods as long as any changes or selected options in the methods are documented and included in the methods manual. (See 10.1.2.)

SOPs – Requirements: Where existing methods are specified as required for a project, requirements contained within that method shall be followed. Any modifications to existing method requirements require project-specific approval by DoD personnel.

SOPs must document complete laboratory-specific instructions regarding equipment, processes, and procedures to a level of detail that would allow a technically qualified individual to repeat the procedure.

While published test methods may be included as part of an SOP, to fulfill the complete requirements of the SOP (as listed in Section 10.1.2.b, items 1-23), it is anticipated that additional information beyond the published test method documentation shall be required.

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c) Copies of all SOPs shall be accessible to all personnel.

d) The SOPs shall be organized.

e) Each SOP shall clearly indicate the effective date of the document, the revision number and the signature(s) of the approving authority.

SOPs – Archiving of SOPs: All SOPs shall be archived for historical reference in accordance with Section 12.1 (Record-Keeping System and Design).

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10.1.2 Laboratory Method Manual(s)

a) The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.

SOPs – Modifications to Existing Methods: Where existing methods are specified as required for a project, requirements contained within that method shall be followed. Any modifications to existing method requirements require project-specific approval by DoD personnel.

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b) This manual may consist of copies of published or referenced test methods or standard operating procedures that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described. Each test method shall include or reference where applicable:

SOPs – Analytical Method SOPs: These requirements apply to all Analytical Method SOPs. While published test methods may be included as part of an SOP, to fulfill the complete requirements of the SOP, as listed immediately below, it is anticipated that additional information beyond the published test method documentation will be required, including, but not limited to:

- Troubleshooting;
- Personnel qualifications;
- Data management and records; and
- Computer hardware and software.

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1) Identification of the test method;
2) Applicable matrix or matrices;
3) Detection limit;
4) Scope and application, including components to be analyzed;
5) Summary of the test method;
6) Definitions;
7) Interferences;
8) Safety;
9) Equipment and supplies;
10) Reagents and standards;
11) Sample collection, preservation, shipment, and storage;
12) Quality control;
13) Calibration and standardization;
14) Procedure;
15) Calculations;
16) Method performance;
17) Pollution prevention;
18) Data assessment and acceptance criteria for quality control measures;
19) Corrective actions for out-of-control data;
20) Contingencies for handling out-of-control or unacceptable data;
21) Waste management;
22) References; and
23) Any tables, diagrams, flowcharts, and validation data.

10.2 Test Methods

The laboratory shall use appropriate test methods and procedures for all tests and related activities within its responsibility (including sample collection, sample handling, transport and storage, sample preparation and sample analysis). The method and procedures shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations or tests concerned.

a) When the use of specific test methods for a sample analysis is mandated or requested, only those methods shall be used.

b) Where test methods are employed that are not required, as in the Performance Based Measurement System approach, the methods shall be fully documented and validated (see 10.2.1 and Appendix C), and be available to the client and other recipients of the relevant reports.

10.2.1 Demonstration of Capability

a) Prior to acceptance and institution of any test method, satisfactory demonstration of method capability is required. (See Appendix C and 6.2.b.) In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (sample of a matrix is which no target analytes or interferences are present at concentrations that impact the results of a

specific test method), e.g., water, solids, biological tissue and air. In addition, for analytes that do not lend themselves to spiking, the demonstration of capability may be performed using quality control samples.

Capability – New Methods Capability: In the case of a laboratory introducing a new method, demonstration of performance shall be determined using an external source of information, when available (for example, the published method). If there is no external source of information, the laboratory shall use comparisons provided by DoD personnel. The laboratory shall not "benchmark against itself" using internal comparisons to initial runs to demonstrate capability.

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b) Thereafter, continuing demonstration of method performance, as per the quality control requirements in Appendix D (such as laboratory control samples) is required.

Capability – Initial and Continuing:~~Method Sensitivity Checks:~~ The initial and continuing demonstration of capability shall include verification of method sensitivity checks (for example, through the use of quarterly method detection verification) and demonstrated measurements of accuracy and precision (such as the production and review of quality control charts). These requirements apply to each matrix of concern.

In addition, continued proficiency (as discussed ~~needed~~ in item c below) shall, at a minimum, include successful analysis of a PT sample by each analyst, annually.

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c) In cases where a laboratory analyzes samples using a test method that has been in use by the laboratory before July 1999, and there have been no significant changes in instrument type, personnel or test method, the continuing demonstration of method performance and the analyst's documentation of continued proficiency shall be acceptable. The laboratory shall have records on file to demonstrate that an initial demonstration of capability is not required.

d) In all cases, the appropriate forms, such as the Certification Statement (Appendix C), must be completed and retained by the laboratory to be made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the Certification Statement must be retained by the laboratory. (See Appendix C for Certification Statement.)

e) A demonstration of capability must be completed each time there is a significant change in instrument type, personnel, or test method.

Capability – Significant Change: "Significant change" refers to any change in personnel, instrumentation, test method, or sample matrix that potentially impacts the precision, accuracy, sensitivity, and selectivity of the output (for example, a change in the detector, column, matrix, or other components of the sample analytical system, or a method revision). Requirements for demonstration of capability are further addressed in Appendix C.

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f) In laboratories with a specialized "work cell(s)" (a group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit must meet the above criteria and this demonstration of capability must be fully documented.

g) When a work cell(s) is employed, and the members of the cell change, the new employee(s) must work with experienced analyst(s) in that area of the work cell where they are employed. This new work cell must demonstrate acceptable performance through acceptable continuing performance checks (appropriate sections of Appendix D, such as laboratory control samples). Such performance must be documented and the four preparation batches following the change in personnel must not result in the

failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. In addition, if the entire work cell is changed/replaced, the work cell must perform the demonstration of capability (Appendix C).

h) When a work cell(s) is employed the performance of the group must be linked to the training record of the individual members of the work cell (See Section 6.2).

Work Cell – Definition of Work Cell: A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation, extraction, and analysis. To ensure that the entire preparation, extraction, and analysis process is completed by a collection of capable individuals, the laboratory shall ensure that **each member** of the work cell (including a new member of an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a "team," the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance.

A work cell may NOT be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

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10.3 Sample Aliquots

Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples.

Sampling – Deviations from Laboratory's Sampling Procedures: Sampling procedures shall also address laboratory practices for the handling, subsampling, and documenting of extraneous materials (for example, rocks, twigs, vegetation) present in samples. The handling of multiphase samples shall be addressed in specific sampling procedures, as appropriate. When a client requires deviations from the laboratory's documented sampling procedure, all deviations shall be recorded in detail in laboratory records and in all test reports. Additionally, the laboratory shall use recognized consensus standards (for example, ASTM standards) where available for these procedures.

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10.4 Data Verification

Calculations and data transfers shall be subject to appropriate checks.

a) The laboratory shall establish Standard Operating Procedures to ensure that the reported data are free from transcription and calculation errors.

b) The laboratory shall establish Standard Operating Procedures to ensure that all quality control measures are reviewed, and evaluated before data are reported.

c) The laboratory shall establish Standard Operating Procedures addressing manual calculations including manual integrations.

Data – Data Verification Procedures: Data verification (review) shall consist of at least the following procedures:

1. Determinations of whether the results of testing, examining, or analyzing the sample meet the laboratory's requirements for interpretation, precision, and accuracy.
2. Checks to determine accuracy of calculations, conversions, and data transfers.
3. Checks for transcription errors, omissions, and mistakes.
4. Checks to determine consistency with project-specific measurement quality objectives (MQOs).
5. Checks to ensure that the appropriate preparatory and analytical SOPs and standardized methods were followed, and that chain-of-custody (COC) and holding time requirements were met.
6. Checks to ensure that requirements for calibration and calibration verification standards were met, and that QC samples (for example, method blanks, laboratory control samples (LCSs)) met criteria for precision, accuracy, and sensitivity.
7. Accurate explanation in the case narrative of any anomalous results and any corrective actions taken, and all data flags checked for appropriate and accurate use.
8. A tiered or sequential system of verification, consisting of at least three levels, with each successive check performed by a different person. This three-tiered approach should include (at a minimum) 100% review by the analyst, 100% verification review by a technically qualified supervisor or data review specialist, and a final administrative review. The final administrative review will verify that previous reviews were documented properly and that the data package is complete.

Additionally, as part of its internal quality assurance program, the quality assurance officer, or designee, shall review at a minimum 10% of all data packages for technical completeness and accuracy. This review is part of the oversight program and does not have to be completed in "real tim

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10.5 Documentation and Labeling of Standards and Reagents

Documented procedures shall exist for the purchase, reception and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory shall retain records for all standards, reagents and media including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material shall not be used unless it is verified by the laboratory.

b) Original containers (such as provided by the manufacturer or vendor) shall be labeled with an expiration date.

c) Records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

Documentation – Lot Number: The records shall include appropriate lot numbers for the standard.

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d) All containers of prepared reagents and standards must bear a unique identifier and expiration date and be linked to the documentation requirements in Section 10.5.c above.

10.6 Computers and Electronic Data Related Requirements

Where computers, automated equipment, or microprocessors are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data, the laboratory shall ensure that:

Electronic Data – Audit Trails: The following applies to audit trails as well as to test data.

In addition to meeting all requirements of this standard (item a below), DoD expects that laboratories employing electronic data processing equipment shall put in place a quality system for such activities that is consistent with the language in Sections 8.1 through 8.11 of the EPA document "2185 – Good Automated Laboratory Practices" (1995). This quality system shall be documented in the laboratory quality manual and appropriate SOPs.

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a) All requirements of this Standard (i.e., Chapter 5 of NELAC) are met;

b) Computer software is tested and documented to be adequate for use, e.g., internal audits, personnel training, focus point of QA and QC;

c) Procedures are established and implemented for protecting the integrity of data; such procedures shall include, but not be limited to, integrity of data entry or capture, data storage, data transmission and data processing;

Data – Automated Processes: At a minimum, for those processes that are automated, a sample data test set shall be used to test and verify the correct operation of these data reduction procedures (including data capture, manipulation, transfer, and reporting). This shall be done anytime new software is purchased or the programming code is modified or otherwise manipulated, and applies even in cases where commercial software is used as part of the process.

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d) Computer and automated equipment are maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data; and

e) It establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

11.0 SAMPLE HANDLING, SAMPLE ACCEPTANCE POLICY AND SAMPLE RECEIPT

While the laboratory may not have control of field sampling activities, the following are essential to ensure the validity of the laboratory's data.

11.1 Sample Tracking

a) The laboratory shall have a documented system for uniquely identifying the items to be tested, to ensure that there can be no confusion regarding the identity of such items at any time. This system shall include identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code to each sample container received in the laboratory. The use of container shape, size, or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.

b) This laboratory code shall maintain an unequivocal link with the unique field ID code assigned each container.

c) The laboratory ID code shall be placed on the sample container as a durable label.

d) The laboratory ID code shall be entered into the laboratory records (see 11.3.d) and shall be the link that associates the sample with related laboratory activities such as sample preparation or calibration.

e) In cases where the sample collector and analyst are the same individual or the laboratory preassigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

11.2 Sample Acceptance Policy

The laboratory must have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted or rejected. Data from any samples that do not meet the following criteria must be flagged in an unambiguous manner, clearly defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collection personnel and shall include, but is not limited to, the following areas of concern:

Sampling – Sample Acceptance: The laboratory shall have procedures documented in the ~~Q~~quality ~~M~~manual or related documentation (as discussed in Sections 5.2.i. and 5.2.k.) that address methods by which the laboratory confirms that it has the capability to accept new samples before such acceptance occurs. The laboratory shall also follow any additional method-specific requirements concerning sample acceptance.

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a) Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

b) Proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) Use of appropriate sample containers;

d) Adherence to specified holding times;

e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests; and

f) Procedures to be used when samples show signs of damage, contamination or inadequate preservation.

11.3 Sample Receipt Protocols

a) Upon receipt, the condition of the sample, including any abnormalities or departures from standard condition as prescribed in the relevant test method, shall be recorded. All items specified in 11.2 above shall be checked.

1) All samples which require thermal preservation shall be considered acceptable if the arrival temperature is either within 2 °C of the required temperature or the method specified range. For samples with a specified temperature of 4 °C, samples with a temperature ranging from just above the freezing temperature of water to 6 °C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

Sampling – Temperature Measurements: The temperature measurement, when applicable, shall be verified through the use of a temperature blank (for each transport container [for example, cooler]) or other measurement when a temperature blank is not available (for example, IR gun).

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2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH or free chlorine, prior to or during sample preparation or analysis.

Sampling – Chemical Preservation of Samples: This shall also be performed when the continued preservation of the sample is in question (due to sample interaction with the preservative); when samples cannot be checked upon receipt (for example, VOCs); and/or for samples whose preservative may have deteriorated for any other reason.

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b) The results of all checks shall be recorded.

c) Where there is any doubt as to the item's suitability for testing, where the sample does not conform to the description provided, or where the test required is not fully specified, the laboratory shall attempt to consult the client for further instruction before proceeding. The laboratory shall establish whether the sample has received all necessary preparation, or whether the client requires preparation to be undertaken or arranged by the laboratory. If the sample does not meet the sample receipt acceptance criteria listed in this standard, the laboratory shall either:

Sampling – Consultation with Client: This consultation shall be immediate and timely (i.e., by the next business day or as specified in project plans).

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1) Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

2) Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

i. The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

ii. The analysis data shall be appropriately "qualified" on the final report.

Sampling – Documentation When Acceptance Criteria Not Met: Additional guidance on this issue is provided in Section 13.a), (Laboratory Report Format and Contents).

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d) The laboratory shall utilize a permanent chronological record such as a log book or electronic database to document receipt of all sample containers.

Data – Electronic Databases: Use of electronic database systems shall meet the requirements specified in Section 10.6., (Computers and Electronic Data-Related Requirements).

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1) This sample receipt log shall record the following:

i. Client/Project Name;

ii. Date and time of laboratory receipt;

iii. Unique laboratory ID code (see 11.1); and

iv. Signature or initials of the person making the entries.

2) During the log-in process, the following information must be unequivocally linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request, and readily

available to individuals who will process the sample. Note: The placement of the laboratory ID number on the sample container is not considered a permanent record.

i. The field ID code that identifies each container must be linked to the laboratory ID code in the sample receipt log.

ii. The date and time of sample collection must be linked to the sample container and to the date and time of receipt in the laboratory.

iii. The requested analyses (including applicable approved test method numbers) must be linked to the laboratory ID code.

iv. Any comments resulting from inspection for sample rejection shall be linked to the laboratory ID code.

e) All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.

f) A complete chain of custody record form (Sections 12.3 and Appendix E), if utilized, shall be maintained.

~~**Sampling – Legal COC and Sample Custody:** Legal COC procedures, as addressed in Section 12.4, shall be required only as specified by DoD project or contract personnel. Standard requirements for sample custody are outlined in Sections 12.1, 12.2, and 12.3 and shall be followed as the default requirement.~~

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11.4 Storage Conditions

The laboratory shall have documented procedures and appropriate facilities to avoid deterioration, contamination, or damage to the sample during storage, handling, preparation, and testing; any relevant instructions provided with the item shall be followed. Where items have to be stored or conditioned under specific environmental conditions, these conditions shall be maintained, monitored, and recorded.

a) Samples shall be stored according to the conditions specified by preservation protocols:

1) Samples which require thermal preservation shall be stored under refrigeration which is +/-2° of the specified preservation temperature unless method specific criteria exist. For samples with a specified storage temperature of 4 °C, storage at a temperature above the freezing point of water to 6 °C shall be acceptable.

Sampling – Refrigerated Samples: When refrigeration or freezing is required, the laboratory shall ensure that monitoring is performed 7 days per week to ensure that the samples remain within an acceptable range. A variety of low cost devices (for example, digital minimum/maximum thermometers with memory, circle chart thermometers) can be used to validate that the proper temperature is continuously maintained.

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2) Samples shall be stored away from all standards, reagents, food, and other potentially contaminating sources. Samples shall be stored in such a manner to prevent cross contamination.

Sampling – Cross Contamination: The laboratory shall have procedures in place to ensure that cross contamination does not occur. Samples designated for volatile organics testing shall be segregated from other samples. Samples suspected to contain high levels of volatile organics shall be further isolated from other volatile organics samples or storage blanks shall be used to verify that no cross contamination has occurred.

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b) Sample fractions, extracts, leachates, and other sample preparation products shall be stored according to 11.4.a above or according to specifications in the test method.

c) Where a sample or portion of the sample is to be held secure (for example, for reasons of record, safety or value, or to enable check calibrations or tests to be performed later), the laboratory shall have storage and security arrangements that protect the condition and integrity of the secured items or portions concerned.

11.5 Sample Disposal

The laboratory shall have standard operating procedures for the disposal of samples, digestates, leachates and extracts or other sample preparation products.

Sampling – Disposal Records: The laboratory shall maintain appropriate documentation and records demonstrating that samples have been properly disposed of, in accordance with Federal, State, and local regulations.

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12.0 RECORDS

The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. The system shall produce unequivocal, accurate records that document all laboratory activities. The laboratory shall retain all original observations, calculations and derived data, calibration records and a copy of the test report for a minimum of five years.

There are two levels of sample handling: 1) sample tracking and 2) legal chain of custody protocols, which are used for evidentiary or legal purposes. All essential requirements for sample tracking (e.g., chain of custody form) are outlined in Sections 12.1, 12.2 and 12.3. If a client specifies that a sample will be used for evidentiary purposes, then a laboratory shall have a written SOP for how that laboratory will carry out legal chain of custody for example, ASTM D 4840-95 and Manual for the Certification of Laboratories Analyzing Drinking Water, March 1997, Appendix A.

~~**Sampling – Legal COC and Sample Custody:** Legal COC procedures, as addressed in Section 12.4, shall be required only as specified by DoD project or contract personnel. Standard requirements for sample custody are outlined in Sections 12.1, 12.2, and 12.3 and shall be followed as the default requirement.~~

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12.1 Record Keeping System and Design

The record keeping system must allow historical reconstruction of all laboratory activities that produced the analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples and/or extracts.

a) The records shall include the identity of personnel involved in sampling, sample receipt, preparation, calibration or testing.

b) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification, shall be documented.

c) The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes, e.g., set format for naming electronic files.

d) All changes to records shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records such as “sampled by,” “prepared b

e) All generated data, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legibly in permanent ink.

f) Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria also shall apply to electronically maintained records.

g) Refer to 10.6 for Computer and Electronic Data.

12.2 Records Management and Storage

a) All records (including those pertaining to calibration and test equipment), certificates and reports shall be safely stored, and held secure and in confidence to the client. NELAP-related records shall be available to the accrediting authority.

b) All records, including those specified in Section 12.3, shall be retained for a minimum of five years from generation of the last entry in the records. All information necessary for the historical reconstruction of data must be maintained by the laboratory. Records which are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

c) Records that are stored or generated by computers or personal computers shall have hard copy or write-protected backup copies.

d) The laboratory shall establish a record management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation storage and reporting.

e) Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

f) The laboratory shall have a plan to ensure that the records are maintained or transferred according to the clients’ instructions (see 4.1.8.e of NELAC) in the event that a laboratory transfers ownership or goes out of business. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed.

12.3 Laboratory Sample Tracking

12.3.1 Sample Handling

A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These shall include but are not limited to all records pertaining to:

a) Sample preservation including appropriateness of sample container and compliance with holding time requirement;

b) Sample identification, receipt, acceptance or rejection and log-in;

c) Sample storage and tracking including shipping receipts, sample transmittal forms (chain of custody form); and

d) The laboratory shall have documented procedures for the receipt and retention of test items, including all provisions necessary to protect the integrity of samples.

12.3.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following shall be retained:

a) All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);

b) A written description or reference to the specific test method used, which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

c) Copies of final reports;

d) Archived standard operating procedures;

e) Correspondence relating to laboratory activities for a specific project;

f) All corrective action reports, audits and audit responses;

g) Proficiency test results and raw data; and,

h) Results of data review, verification, and cross-checking procedures.

12.3.3 Analytical Records

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, shall include:

a) Laboratory sample ID code;

b) Date of analysis and time of analysis is required if the holding time is 72 hours or less or when time critical steps are included in the analysis, e.g., extractions, and incubations;

Analytical Records – Time of Analysis: For DoD work, both date and **time** of analysis are considered to be essential information, regardless of the length of the holding time, and shall be included as part of the analytical record.

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c) Instrumentation identification and instrument operating conditions/parameters (or reference to such data);

d) Analysis type;

e) All manual calculations e.g., manual integrations;

f) Analyst's or operator's initials/signature;

g) Sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

h) Sample analysis;

i) Standard and reagent origin, receipt, preparation, and use;

j) Calibration criteria, frequency and acceptance criteria;

k) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

l) Quality control protocols and assessment;

m) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and

n) Method performance criteria including expected quality control requirements.

12.3.4 Administrative Records

The following shall be maintained:

a) Personnel qualifications, experience and training records;

b) Records of demonstration of capability for each analyst; and

c) A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

13.0 LABORATORY REPORT FORMAT AND CONTENTS

The results of each test, or series of tests carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively. The results shall normally be reported in a test report and shall include all the information necessary for the interpretation of the test results and all information required by the method used. Some regulatory reporting requirements or formats, such as monthly operating reports may not require all items listed below, however, the laboratory shall provide all the required information to their client for use in preparing such regulatory reports.

Reporting Requirements: The reporting requirements for work produced for DoD are outlined in Appendix DoD-A. This appendix follows all the NELAC appendices.

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a) Except as discussed in 13.b, each report to an outside client shall include at least the following information (those prefaced with “where relevant” are not mandatory):

1) A title, e.g., "Test Report," or "Test Certificate," "Certificate of Results" or "Laboratory Results”;

2) Name and address of laboratory, and location where the test was carried out if different from the address of the laboratory and phone number with name of contact person for questions;

3) Unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;

This requirement may be presented in several ways:

i. The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or

ii. Each page is identified with the unique report identification, the pages are identified as a number of the total report pages (example: 3 of 10, or 1 of 20).

Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report, and that the report contains a specified number of pages.

4) Name and address of client, where appropriate and project name if applicable;

5) Description and unambiguous identification of the tested sample including the client identification code;

6) Identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;

7) Date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 72 hours;

Laboratory Report Contents – Time of Analysis: For DoD work, both date and **time** of analysis are considered to be essential information, regardless of the length of the holding time, and shall be included as part of the laboratory report.

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8) Identification of the test method used, or unambiguous description of any nonstandard method used;

9) If the laboratory collected the sample, reference to sampling procedure;

10) Any deviations from (such as failed quality control), additions to or exclusions from the test method (such as environmental conditions), and any nonstandard conditions that may have affected the quality of results, and including the use and definitions of data qualifiers.

11) Measurements, examinations and derived results, supported by tables, graphs, sketches, and photographs as appropriate, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis; identify the reporting units such as µg/l or mg/kg; and for Whole Effluent Toxicity, identify the statistical package used to provide data;

12) When required, a statement of the estimated uncertainty of the test result;

13) A signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the certificate or report (however produced), and date of issue;

14) At the laboratory's discretion, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory;

15) At the laboratory's discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;

16) Clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc.; and

17) Clear identification of numerical results with values outside of quantitation limits.

b) Laboratories that are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes (in-house or captive laboratories) shall have all applicable information specified in 1 through 17 above readily available for review by the accrediting authority. However formal reports detailing the information are not required if:

1) The in-house laboratory is itself responsible for preparing the regulatory reports; or

2) The laboratory provides information to another individual within the organization for preparation of regulatory reports. The facility management must ensure that the appropriate report items are in the report to the regulatory authority if such information is required.

c) Where the certificate or report contains results of tests performed by subcontractors, these results shall be clearly identified by subcontractor name or applicable accreditation number.

d) After issuance of the report, the laboratory report shall remain unchanged. Material amendments to a calibration certificate, test report or test certificate after issue shall be made only in the form of a further document, or data transfer, including the statement "Supplement to Test Report or Test Certificate, serial number . . . [or as otherwise identified]", or equivalent form of wording. Such amendments shall meet all the relevant requirements of this Standard.

e) The laboratory shall notify clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any calibration certificate, test report or test certificate or amendment to a report or certificate.

f) The laboratory shall, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, follow documented procedures that ensure that the requirements of this Standard are met and that confidentiality is preserved.

g) Laboratories accredited to be in compliance with these standards shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

~~**Quality Manual – Supplemental Manuals:** As noted in the DoD Introduction to this document, DoD plans to supplement this Mmanual with other standardized documents and formats to support and unify the laboratory analysis and reporting process. It is anticipated that a standardized laboratory report format will be issued as part of this continuing effort. In the meantime, there may be additional component-specific or project-specific requirements that supplement those listed above.~~

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14.0 SUBCONTRACTING ANALYTICAL SAMPLES

a) The laboratory shall advise the client in writing of its intention to subcontract any portion of the testing to another party.

b) Where a laboratory subcontracts any part of the testing covered under NELAP, this work shall be placed with a laboratory accredited under NELAP for the tests to be performed.

c) The laboratory shall retain records demonstrating that the above requirements have been met.

15.0 OUTSIDE SUPPORT SERVICES AND SUPPLIES

a) Where the laboratory procures outside services and supplies, other than those referred to in this Standard, in support of tests, the laboratory shall use only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests.

b) Where no independent assurance of the quality of outside support services or supplies is available, the laboratory shall have procedures to ensure that purchased equipment, materials and services comply with specified requirements. The laboratory shall ensure that purchased equipment and consumable materials are not used until they have been inspected, calibrated or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned.

~~**Materials Handling:** The laboratory **shall** ensure that materials are inspected, calibrated, or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned.~~

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c) The laboratory shall maintain records of all suppliers from whom it obtains support services or supplies required for tests.

Supplier Records: These records shall include date of receipt, expiration date (where applicable), source (i.e., provider or supplier), lot number, and calibration and verification records and certifications for whatever supplies and services may impact the usability of associated test results. Examples of these materials that may have an impact on the quality of data include solvents, standards, Class A glassware, and sample containers. Furthermore, all of these supplies shall be maintained according to the applicable requirements specified in Sections 9.3 and 10.5.

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16.0 COMPLAINTS

The laboratory shall have documented policy and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities. Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this Standard or otherwise concerning the quality of the laboratory's calibrations or tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with Section 5.3.1. Records of the complaint and subsequent actions shall be maintained.

Complaints/Problems Response System: The laboratory's quality system shall contain a process for responding to complaints and/or problems. At a minimum, this will include tracking of quality checks, internal audits, and quality control trending. Documentation of this response and resolution of the problem, as applicable to DoD, shall be maintained. In addition, the laboratory shall use this information as part of its quality system to identify patterns of problems and to correct them. These logs shall be available for DoD review, to help DoD assess the effectiveness of the laboratory's corrective action process. This information will be considered to be confidential, but will, nonetheless, be used by DoD to assess the effectiveness of the laboratory's quality system.

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NELAC APPENDICES

APPENDIX A - REFERENCES

40 CFR Part 136, Appendix A, paragraphs 8.1.1 and 8.2.

American Association for Laboratory Accreditation April 1996. General Requirements for Accreditation.

"American National Standards Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4)," 1994.

ASTM E1598-94 Conducting Early Seedling Growth Tests, American Society for Testing and Materials, West Conshohocken, PA 1999.

ASTM E11676-97 Conducting a Laboratory Soil Toxicity Test with Lumbricid Earthworm *Eisenia foetida*, American Society for Testing and Materials, West Conshohocken, PA 1999.

Catalog of Bacteria, American Type Culture Collection, Rockville, MD.

EPA 2185 - Good Automated Laboratory Practices, 1995 available at www.epa.gov/docs/etsdwe1/irm_galp/

EPA/600/3-89/013 Ecological Assessment of Hazardous Waste Sites, Office of Research and Development, Washington, DC, 1991.

EPA/503/8-91/001 Evaluation of Dredged Material Proposed for Ocean Disposal – Testing Manual. Office of Water, Washington, DC, 1991.

EPA/600/4-90/031 Manual for Evaluation of Laboratories Performing Aquatic Toxicity Tests, Office of Research and Development, Washington, DC, 1991.

EPA/600/3-88/029 Protocol for Short-term Toxicity Screening of Hazardous Wastes, Office of Research and Development, Washington, DC, 1991.

EPA/600/4-90/027F Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, 4th Ed., Office of Research and Development, Washington, DC, 1993.

EPA/823/B-98/004 Evaluation of Dredged Material Proposed for Discharge in Waters of the U.S. – Inland Testing Manual. Office of Water, Washington, DC, 1994.

EPA/600/R-94/025 Methods for Assessing the Toxicity of Sediment-associated Contaminants with Estuarine and Marine Amphipods, Office of Research and Development, Washington, DC, 1994.

EPA/600/R-94/024 Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates, Office of Research and Development, Washington, DC, 1994.

EPA/600/4-91/002 Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, 3rd Ed., Office of Research and Development, Washington, DC, 1994.

EPA/600/4-91/003 Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Water to Marine and Estuarine Organisms, 2nd Ed., Office of Research and Development, Washington, DC, 1994.

"Glossary of Quality Assurance Terms and Acronyms," Quality Assurance Division, Office of Research and Development, USEPA.

"Guidance on the Evaluation of Safe Drinking Water Act Compliance Monitoring Results from Performance Based Methods," September 30, 1994, Second draft.

International vocabulary of basic and general terms in metrology (VIM): 1984. Issued by BIPM, IEC, ISO and OIML.

ISO Guide 3534-1: "Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms."

ISO Guide 7218: Microbiology - General Guidance for Microbiological Examinations.

ISO Guide 8402: 1986. Quality – Vocabulary.

ISO Guide 9000: 1994. Quality management and quality assurance standards - Guidelines for selection and use.

ISO Guide 9001: 1994. Quality Systems - Model for quality assurance in design/development, production, installation and servicing

ISO Guide 9002: 1994. Quality systems - Model for quality assurance in production and installation.

ISO/IEC Guide 2: 1986. General terms and their definitions concerning standardization and related activities.

ISO/IEC Guide 25: 1990. General requirements for the competence of calibration and testing laboratories.

"Laboratory Biosafety Manual," World Health Organization, Geneva, 1983.

Manual for the Certification of Laboratories Analyzing Drinking Water, Revision 4, EPA 815-B-97-001.

Manual of Method for General Bacteriology, Philipp Gerhard et al., American Society for Microbiology, Washington, D.C. 1981.

NELAC Standards, Chapters 1-6. June 29, 2000.

Performance Based Measurement System, EPA EMMC Method Panel, PBMS Workgroup, 1996.

APPENDIX B - GLOSSARY

The following definitions are used in the text of Quality Systems. In writing this document, the following hierarchy of definition references were used: ISO 8402, ANSI/ASQC E-4, EPA's Quality Assurance Division Glossary of Terms, and finally definitions developed by NELAC. The source of each definition, unless otherwise identified, is the Quality Systems Committee.

Quality Systems Definitions: The Quality Systems Committee is the NELAC-appointed group that created and continues to modify NELAP Chapter 5 (Quality Systems). Terms not included in the NELAC Glossary, but defined by DoD, are included in gray text boxes throughout this Appendix.

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Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority: The Territorial, State, or Federal agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation. (NELAC) [1.5.2.3]

Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (Team, EPA QAD Glossary)

Analysis Duplicate: The second measurement of the target analyte(s) performed on a single sample or sample preparation.

Analyst: The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

Analyte: The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)

Analytical Detection Limit: The smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (Applicable only to radiochemistry)

Analytical Reagent (AR) Grade: Designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society. (Quality Systems)

Assessment: The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

Audit: A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

Batch: Environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

Blank: A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample: A sub-sample for analysis with a composition known to the submitter. The analyst/ laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process. (NELAC)

Calibration: To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method: A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard: A substance or reference material used to calibrate an instrument. (QAMS)

Certified Reference Material (CRM): A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody Form: A record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC)

Chemical: Any element, compound, or mixture of elements and/or compounds. Frequently, chemical substances are classified by the CAS rules of nomenclature for the purposes of identification for a hazard evaluation. (OSHA Glossary)

Client: The party that has agreed to pay the bill for services rendered by the laboratory, and with whom the laboratory has a contractual relationship for that project. For a laboratory, this is typically the prime contractor who originally hires the laboratory for the project, and who signs the contract as the receiver of services and resulting data. In cases where the laboratory has a direct contractual relationship with DoD, the client shall be the Government's authorized contracting officer. The contracting officer, as the client, shall consult with the Government's authorized technical representative when dealing with laboratory technical issues. It is understood that typically other "Clients" are present at other levels of the project, but they may be removed from the day-to-day decision-making (for example, installation representatives, service center representatives, various other Government officials). Specific circumstances may require the direct notification of these other clients, in addition to the prime contractor or DoD representative; these circumstances shall be included as part of specific project requirements. (Team)

Compound: A unique combination of chemical elements, existing in combination to form a single chemical entity. (Team)

Component: A single chemical entity, such as an element or compound. Multiple components may compose one analyte. (OSHA Glossary, Team)

Compromised Samples: Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions compromised samples are not analyzed. If emergency situations require analysis, the results must be appropriately qualified. (NELAC)

Confirmation: Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

- Second column confirmation;
- Alternate wavelength;
- Derivatization;
- Mass spectral interpretation;
- Alternative detectors; or
- Additional cleanup procedures. (NELAC)

Conformance: An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ ASQC E4-1994)

Consensus Standards: A protocol established by a recognized authority (for example, American Society for Testing and Materials [ASTM], American National Standards Institute [ANSI], or the Institute for Electrical and Electronic Engineers [IEEE]).

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

Data Reduction: The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Definitive Data: Data that are generated using rigorous analytical methods, such as approved EPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce tangible raw data in the form of paper printouts or electronic files. Data shall satisfy QA/QC requirements. For data to be definitive, either analytical or total measurement error shall be determined and documented. (Data Quality Objectives Process for Superfund)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate acceptable accuracy. (NELAC)

Desorption Efficiency: The mass of target analyte recovered from sampling media, usually a sorbent tube, divided by the mass of target analyte spiked on to the sampling media expressed as a percentage. Sample target analyte masses are usually adjusted for the desorption efficiency. (NELAC)

Detection Limit: The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA- QAD)

Environmental Program: An organized effort that assesses environmental concerns and leads to the collection of data, either in the field or through laboratory analysis. (Variation on EPA QAD Glossary for Terms: Environmentally related measurement, environmental sample)

Holding Times (Maximum Allowable Holding Times): The maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR Part 136)

Holding Times (DoD Clarification): The time elapsed from the time of sampling to the time of extraction or analysis, as appropriate.

Inspection: An activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ ASQC E4-1994)

Internal Standard: A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method. (NELAC)

Instrument Blank: A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Key Staff: At a minimum, the following managerial and supervisory staff (however named) – executive staff (for example, Chief Executive Officer, Chief Operating Officer, laboratory director, technical director); technical directors/supervisors (for example, section supervisors for organics and inorganics); quality assurance systems directors/supervisors (for example, QA officer, quality auditors); and support systems directors/supervisors (for example, information systems supervisor, purchasing director, project manager).

Laboratory: A body that calibrates and/or tests. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC).

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Limit of Detection (LOD): The lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. See also Method Detection Limit, Detection Limit, and Quantitation Limit (Analytical Chemistry, 55, p. 2217, December 1983, modified)

Manager (however named): The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual. (NELAC)

Matrix: The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

- Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.
- Drinking Water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous Liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.
- Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
- Air: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample): A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (QAMS)

May: Denotes permitted action, but not required action. (NELAC)

Media: Material that supports the growth of a microbiological culture.

Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

Method Detection Limit: The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B)

Must: Denotes a requirement that must be met. (Random House College Dictionary)

National Accreditation Database: The publicly accessible database listing the accreditation status of all laboratories participating in NELAP. (NELAC)

National Environmental Laboratory Accreditation Conference (NELAC): A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

National Environmental Laboratory Accreditation Program (NELAP): The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Negative Control: Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

Nonconformance: An indication or judgment that a product or service has not met the requirements of the relevant specifications, contract or regulation; also the state of failing to meet the requirements.

Objective Evidence: Any documented statement of fact, other information, or record, either quantitative or qualitative, pertaining to the quality of an item or activity, based on observations, measures, or tests that can be verified. (ASQC)

Performance Audit: The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS): A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision: The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

Proficiency Testing: A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

Proficiency Testing Program: The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

Proficiency Test Sample (PT): A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

Protocol: A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed. (EPA- QAD)

Pure Reagent Water: Shall be water (defined by national or international standard) in which no target analytes or interferences are detected as required by the analytical method. (NELAC)

Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

Quality Assurance (Project) Plan (QAPP): A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)

Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

Quality Control Sample: An uncontaminated sample matrix with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

Quality Manual: A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

Quality System: A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ ASQC E-41994)

Quantitation Limits: Levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported at a specific degree of confidence. (NELAC)

Quantitation Limits (DoD Clarification): The value at which an instrument can accurately measure an analyte at a specific concentration (i.e., a specific numeric concentration can be quantified). These points are established by the upper and lower limits of the calibration range.

Range: The difference between the minimum and the maximum of a set of values. (EPA-QAD)

Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted. (EPA-QAD)

Reagent Blank (method reagent blank): A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions. (EPA-QAD)

Reference Material: A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30- 2.1)

Reference Method: A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

Reference Standard: A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.08)

Reference Toxicant: The toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results (see Chapter 5, Appendix D, Section 2.1.f). (NELAC)

Replicate Analyses: The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

Reporting Limit: ~~A specific concentration at or above the lower quantitation limit that is reported to the client with confidence. It is often defined on a project-specific basis.~~ A data value specified by the client based on sensitivity requirements from project-specific action levels. If initially set by the client below the laboratory's lower quantitation limit, method modification is required or the client will be required to accept the laboratory's lower quantitation limit as the lowest technically valid value that can be provided by the laboratory. For methods that require only one standard and a blank, ~~(for example, lower limit of calibration curve is the origin),~~ a low-level check standard shall be required to establish the lower quantitation limit. ~~t~~The reporting limit shall be no lower than this value~~the low-level check standard~~. Note: There may be numbers reported to the client that are below the reporting limit. These numbers must be flagged appropriately. When the analysis demonstrates a non-detect at the MDL, the data shall be flagged with a "U." The value reported to the client is the MDL, adjusted by any dilution factor used in the analysis. When an analyte is detected between the lower quantitation limit and the MDL, the data shall be flagged with a "J." The value reported is an estimation.

Requirement: Denotes a mandatory specification; often designated by the term "shall". (NELAC)

Sample: Portion of material collected for chemical analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.

Sampling Media: Material used to collect and concentrate the target analytes(s) during air sampling such as solid sorbents, filters, or impinger solutions.

Selectivity: (Analytical chemistry) The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)

Sensitivity: The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

Shall: Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (ANSI)

Should: Denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI)

Spike: A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

Standard Operating Procedure (SOP): A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

Standardized Reference Material (SRM): A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

Supervisor (however named): The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS)

Systems Audit (also Technical Systems Audit): A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Technical Director: Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Testing Laboratory: Laboratory that performs tests. (ISO/ IEC Guide 2 - 12.4)

Test Sensitivity/Power: The minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis (see Chapter 5, Appendix D, Section 2.4.a). (NELAC)

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma) (applies to radiobioassay laboratories). (ANSI)

Traceability: The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Tune – An injected standard required by the method as a check on instrument performance for mass spectrometry.

Validation: The process of substantiating specified performance criteria. (EPA- QAD)

Verification: Confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Work Cell: A well defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)

Sources:

American Society for Quality Control (ASQC), Definitions of Environmental Quality Assurance Terms, 1996

American National Standards Institute (ANSI), Style Manual for Preparation of Proposed American National Standards, Eighth Edition, March 1991

ANSI/ASQC E4, 1994

ANSI N42.23- 1995, Measurement and Associated Instrument Quality Assurance for Radiobioassay Laboratories

International Standards Organization (ISO) Guides 2, 30, 8402

International Vocabulary of Basic and General Terms in Metrology (VIM): 1984. Issued by BIPM, IEC, ISO and OIML

National Institute of Standards and Technology (NIST)

National Environmental Laboratory Accreditation Conference (NELAC), July 1998 Standards

Random House College Dictionary

U.S. EPA Quality Assurance Management Section (QAMS), Glossary of Terms of Quality Assurance Terms, 8/31/92 and 12/6/95

U.S. EPA Quality Assurance Division (QAD)

40 CFR Part 136

Webster’s New World Dictionary of the American Language

APPENDIX C - DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR DEMONSTRATION OF CAPABILITY

A demonstration of capability (DOC) must be made prior to using any test method, and at any time there is a change in instrument type, personnel or test method. (See 10.2.1.)

Capability – Significant Change: "Significant change" refers to any change in personnel, instrumentation, test method, or sample matrix that potentially impacts the precision, accuracy, sensitivity, and selectivity of the output (for example, a change in the detector, column, or other components of the sample analytical system, or a method revision). All new analysts, regardless of experience on that instrument in another laboratory, shall complete a demonstration of capability.

C-1

Note: In laboratories with specialized "work cells" (a well-defined group of analysts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

Work Cell – Definition of Work Cell: Additional guidance on this issue is provided in Section 10.2.1.f. A "work cell" is considered to be all those individuals who see a sample through the complete process of preparation, extraction, and analysis. To ensure that the entire preparation, extraction, and analysis process is completed by a collection of capable individuals, the laboratory shall ensure that **each member** of the work cell (including a new member of an already existing work cell) demonstrates capability in his/her area of responsibility in the sequence. Even though the work cell operates as a "team," the demonstration of capability at each individual step in the sequence, as performed by each individual analyst/team member, remains of utmost importance.

A work cell may NOT be defined as a group of analysts who perform the same step in the same process (for example, extractions for Method 8270), represented by one analyst who has demonstrated capability for that step.

C-2

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids, biological tissue and air. However, before any results are reported using this method, actual sample spike results may be used to meet this standard, i.e., at least four consecutive matrix spikes within the last twelve months. In addition, for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations shall be documented through the use of the form in this appendix.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, shall be performed if required by mandatory test method or regulation. Note: For analytes for which spiking is not an option and for which quality control samples are not readily available, the 40 CFR approach is one way to perform this demonstration. It is the responsibility of the laboratory to document that other approaches to DOC are adequate, and this shall be documented in the laboratory's Quality Manual, e.g., for Whole Effluent Toxicity Testing see section D.2.1.a.1.

a) A quality control sample shall be obtained from an outside source. If not available, the QC sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

c) At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, calculate the mean recovery ($\overline{X}$) in the appropriate reporting units (such as µg/L) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest. When it is not possible to determine mean and standard deviations, such as for presence/absence and logarithmic values, the laboratory will assess performance against established and documented criteria.

Capability – New Methods Evaluation: In the case where the laboratory is introducing a new method, these criteria shall be determined using an external source of information when available (for example, the published method). If there is no external source of information, the laboratory shall use comparisons provided by DoD personnel. The laboratory shall not "benchmark against itself" by using internal comparisons to initial runs to establish these criteria.

C-3

e) Compare the information from (d) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are no established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.

1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.

2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

C.2 CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee (see 6.3 and 12.3.4.b.).

Capability – Certification Statement: All repeated incidences of testing to meet a demonstration of capability shall be documented and packaged with the final certification statement.

C-4

Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix: _____
(examples: laboratory pure water, soil, air, solid, biological tissue)

Method number, SOP#, Rev #, and Analyte, or Class of Analytes or Measured Parameters:
_____ (examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was performed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

_____	_____	_____
Technical Director's Name and Title	Signature	Date
_____	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

APPENDIX D - ESSENTIAL QUALITY CONTROL REQUIREMENTS

DoD Quality Control Requirements: Appendix DoD-B contains tables that consolidate DoD data quality requirements for common SW-846 methods into an easy-to-use reference format. In addition, introductory material identifies definitions of QC checks and clarifies DoD's interpretation of method requirements. This appendix follows all the NELAC appendices.

D-1

The quality control protocols specified by the laboratory's method manual (10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals.

All quality control measures shall be assessed and evaluated on an ongoing basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter 5, e.g., Section 5.4, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing, W.E.T. testing, microbiology testing, radiochemical testing and air testing.

Quality Control – Corrective Action: When quality control measures fail the acceptance criteria specified in these requirements, corrective action shall be taken. Different corrective responses may be appropriate in different situations, based on project-specific requirements and the magnitude of the problem. Examples of corrective actions include:

- Determining the source of the problem,
- Notifying the client,
- Reprocessing samples,
- Using data qualifiers to "flag" data, and
- Adding commentary in laboratory reports.

D-21

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

Target Analyte Lists: The laboratory shall analyze those analytes identified by the client on a project-specific basis. If project-specific information is not available or is incomplete, then the target analyte lists in Appendix DoD-C shall be used. This appendix follows all the NELAC appendices.

D-3

a) Negative Controls

1) Method Blanks - Shall be performed at a frequency of one per preparation batch of samples per matrix type. The results of this analysis shall be one of the QC measures to be used to assess the batch. The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if

i) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch or

ii) the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

Method Blanks: The following paragraphs restate the requirements of Section D.1.1.a.1 above, with DoD expectations with respect to the requirement highlighted in bold.

Method Blanks - Shall be performed at a frequency of one per ~~**preparatory**~~ preparation batch of samples per matrix type **per sample extraction or preparation method**. The results of this analysis shall be one of the QC measures to be used to assess the batch ~~acceptance~~. The source of **method blank** contamination **shall** be investigated~~,~~ and measures taken to correct, minimize~~,~~ or eliminate the problem if **the concentration exceeds one-half the reporting limit. If one-half the reporting limit [RL] is exceeded, the laboratory shall evaluate whether reprocessing of the samples is necessary, based on the following criteria:**

i) The blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated **preparation~~ry~~** batch~~,~~ or
ii) The blank contamination is greater than 1/10 of the specified regulatory limit.

Any samples associated with a blank that fail these criteria checks shall be reprocessed in a subsequent preparation~~ry~~ batch, except when the sample analysis resulted in a nondetect. If no sample volume remains for reprocessing, the results **shall be** reported with appropriate data qualifying codes.

Applicable when method-specific guidance does not exist.

D-~~4~~2

b) Positive Controls

1) Laboratory Control Sample (LCS) - (QC Check Samples) Shall be analyzed at a minimum of 1 per preparation batch of 20 or less samples per matrix type, except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to assess the batch. NOTE: The matrix spike (see 2 below) may be used in place of this control as long as the acceptance criteria are as stringent as for the LCS.

Laboratory Control Sample (LCS): ~~The LCS shall, at a minimum, meet limits specified in the method, if available. In addition, the laboratory shall establish its own limits, based on in-house statistical analysis of historical LCS results. The acceptability of LCS results within any preparatory batch shall be based on these in-house limits, unless the method-specified limits are more stringent, or the client has specified limits based on the intended use of the data.~~ DoD has established LCS control limits based on a multi-laboratory study. The acceptability of LCS results within a preparatory batch shall be determined using these DoD limits or limits specified by the client based on the intended use of the data. The procedures for application of these limits allow for a specific number of sporadic marginal exceedances for some analytical methods. (See Appendix DoD-D for further explanation. This appendix follows all the NELAC appendices.) If DoD limits are not available for certain analytes, the laboratory shall establish its own limits, based on in-house statistical analysis of historical LCS results, and base LCS acceptability on these in-house limits. At a minimum these limits shall meet the limits specified in the method, if available.

D-~~5~~3

2) Matrix Spikes (MS) - Shall be performed at a frequency of one out of every 20 samples per matrix type prepared over time, except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

Matrix Spike Frequency: Matrix spikes shall be performed at a frequency of 1 in 20 samples per matrix type, if adequate sample material is provided by the field investigation. If adequate sample material is not available, then the frequency of matrix spikes shall be noted in the case narrative. Additional matrix spikes may be required by project-specific needs for quality control.

D-64

3) Surrogates - Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

4) If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene, and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses, permit-specified analytes, and other client-requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.

Spiking Compounds:

- The protocols above shall only be required if the test method **or project-specific requirements** do not specify the spiking compounds.
- For DoD, all target analytes must be spiked in the positive control samples (i.e., LCS, MS, MSD). For evaluation and acceptance criteria see Appendices DoD-B and DoD-D.
- The list of "reportable components" is specified by the project.
~~For DoD, "an extremely long list of components" means greater than 50 components reported per method. The exception does not apply to generalized analyte lists (for example, Appendix IX). If a percentage of the component list is used, those analytes must be representative of each chemical class covered by the test method and include any project-specific analytes of concern.~~
- The concentration of the ~~matrix~~ spiked compounds shall be at or below the midpoint of the calibration range.

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D.1.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates - Shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document its procedure to select the use of appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

Matrix Spike Duplicates: Each duplicate named above shall be analyzed using the same specifications as its respective matrix spike. For example, matrix spike duplicates shall be performed at a frequency of 1 in 20 samples per matrix type. Additional matrix spike duplicates may be required by project-specific needs.

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D.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Analytical Capability - (Section 10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

Capability – Significant Change: "Significant change" refers to any change in personnel, instrumentation, test method, or sample matrix that potentially impact the precision and accuracy, sensitivity, and selectivity of the output (for example, a change in the detector, column, or other components of the sample analytical system, or a method revision). Requirements for a demonstration of capability are further addressed in Appendix C.

D-97

b) Calibration - Calibration protocols specified in Section 9.4 shall be followed.

Calibration Protocols: Protocols in Section 9.4 shall be followed, unless method or project-specific procedures and criteria are available.

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c) Proficiency Test Samples - The results of such analyses (4.2.j or 5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

Proficiency Testing: Proficiency testing is discussed further in NELAP Chapter 2. If such testing reveals inaccuracies in data generation, corrective action shall be taken in accordance with the laboratory's documented procedures. DoD shall submit its own proficiency testing samples, as it deems necessary.

D-119

D.1.4 Detection Limits

The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the mandated test method or applicable regulation, e.g., Method Detection Limit (MDL). If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

Detection Limits: A method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from analysis of a sample in a given matrix containing the analyte.

Requirements established in 40 CFR 136B are the baseline source of information for determining MDLs. Other published statistical methods may be appropriate as supplemental resources in determining MDLs (for example, Hubaux and Vos studies may be appropriate for methods that do not require preparation, such as GC/MS volatiles in water). The following list clarifies and expands on the fundamental requirements and principles outlined in 40 CFR 136B, and shall be followed when performing work for DoD:

- As stated in 40 CFR 136B, MDLs shall be determined using a minimum of seven replicates. If more than seven replicates are processed, data cannot be excluded, unless exclusion is supported with sound, documented, technically based justification.
- MDLs are to be calculated for each analyte and matrix. If multiple instruments with identical configurations are used in the laboratory, then the laboratory shall conduct an MDL study on at least one of the instruments and confirm the attainability of that MDL on all instruments by using an MDL verification check sample.
- If multiple MDL results are generated from multiple instruments with identical configurations, then the highest MDL among those may be used in reporting data from all of those instruments. If a lower MDL is reported for specific samples, then the samples must have been run on that specific instrument on which the lower MDL was generated.
- MDLs shall be generated for all applicable matrices, using, at a minimum, a purified matrix free of the analytes of interest (for example, Ottawa sand, reagent-grade water). For metals, teflon chips can be used to simulate the soil matrix.
- MDLs shall be generated for all preparatory and cleanup methods routinely used on samples.
- An MDL verification check shall always be performed immediately following an MDL study. DoD requires that the MDL check sample be spiked at **approximately 2 times** the current reported MDL.
- If an annual MDL study is not performed, MDL verification checks shall be performed **quarterly**. If the quarterly MDL verification check fails, additional MDL verification checks shall be performed at a higher level to set a higher MDL, or the MDL study shall be reconducted.
- For DoD, the MDL verification check sample shall be acceptable if it produces a response that lies at least 3 times above the instrument's noise level.
- Deviations from the above are permitted with the approval of DoD personnel.

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a) A detection limit study is not required for any component for which spiking solutions or quality control samples are not available such as temperature.

b) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).

c) Detection limits must be determined each time there is a change in the test method that affects how the test is performed, or when a change in instrumentation occurs that affects the sensitivity of the analysis.

d) All sample processing steps of the analytical method shall be included in the determination of the detection limit.

e) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.

f) The laboratory must have established procedures to relate detection limits with quantitation limits.

g) The test method's quantitation limits must be established and must be above the detection limits.

Lower Quantitation Limit Establishment: The lower quantitation limit is established by the low standard of the initial calibration curve or the low-level calibration check standard. At a minimum the quantitation limit shall be three times the detection limit.

In addition, in the cases of compounds that are identified by a recognizable pattern (for example, PCBs, toxaphene, technical-chlordane), the quantitation limit is not based solely on the detection limit of the various components, but on the concentration of the mixture at which the pattern becomes recognizable to the analyst.

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D.1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

Data Reduction Procedures – Automated Processes: At a minimum, for those processes that are automated, a sample data test set shall be used to test and verify the correct operation of these data reduction procedures (including data capture, manipulation, transfer, and reporting). This shall be done anytime the programming code is modified or otherwise manipulated and applies even in cases where commercial software is used as part of the process.

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D.1.6 Quality of Standards and Reagents

a) The source of standards shall comply with 9.2.

b) Reagent Quality, Water Quality and Checks:

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

2) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.

SOPs – Water Quality in Method SOPs: When water quality is not specified in the method, the default water quality shall be specified in the method-specific SOPs (for example, ASTM Type I or II) and be of known, documented, and appropriate quality.

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3) The laboratory will verify the concentration of titrants in accordance with written laboratory procedures.

D.1.7 Selectivity

a) Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory shall develop and document acceptance criteria for retention time windows.

Retention Time Verification – Frequency and Criteria: The laboratory shall follow method-specific requirements for frequency of retention time verification and criteria for acceptance. If method-specific requirements do not exist, the laboratory shall develop and document the frequency of retention time verification and the acceptance criteria for retention time windows.

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b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

Data – Data Confirmation: This requirement may be waived by the client in the case of periodic monitoring of well-characterized media that are tested by the same laboratory. For data that are required to be confirmed, all results shall be reported as confirmed or unconfirmed. If unconfirmed data are reported, they shall be identified separately in the report, with a narrative explaining why the data were not confirmed. Evaluation criteria for the confirmation of results shall be as specified by the method, unless otherwise specified by DoD personnel. If method-specific requirements do not exist, the laboratory shall develop and document acceptance criteria for the confirmation of results.

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c) The laboratory shall document acceptance criteria for mass spectral tuning.

Mass Spectral Tuning – Acceptance Criteria: These acceptance criteria are specified by the method, unless otherwise specified by DoD personnel.

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D.1.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

D.2 TOXICITY TESTING

These standards apply to laboratories measuring the toxicity and/or bioaccumulation of contaminants in general. They are applicable to toxicity or bioaccumulation test methods for evaluating effluents (whole effluent toxicity or WET), receiving waters, sediments, elutriates, leachates and soils. In addition to the essential quality control standards described below, some methods may have additional or other requirements based on factors such as the type of matrix evaluated. Additional information can be found in the following methods manuals (or most recent edition): EPA/600/4-91/002, EPA/600/4-91/003, EPA/600/4-90/027F (WET testing), EPA/600/4-90/031 (general aquatic toxicity testing), EPA/600/R-94/025, EPA/600/R-94/024, EPA/503/R-91/001, EPA/823/B-98/004 (sediments and elutriates), EPA/600/3-88/029, EPA/600/3-89/013, ASTM E1598-94 AND ASTM 1676-97 (soils).

D.2.1 Positive and Negative Controls

a) Positive Control - Reference Toxicants - Reference toxicant tests indicate the sensitivity of the test organisms being used and demonstrate a laboratory's ability to obtain consistent results with the test method.

1) The laboratory must demonstrate its ability to obtain consistent results with reference toxicants before it performs toxicity tests with effluents or other environmental samples for regulatory compliance purposes.

   i) To meet this requirement, the intra-laboratory precision must be determined by performing five or more acceptable reference toxicant tests for each test method and species with different batches of organisms and appropriate negative controls (water, sediment, or soil).

   ii) An intralaboratory coefficient of variation (%CV) is not established for each test method. However, a testing laboratory shall maintain control charts for the control performance and reference toxicant statistical endpoint (such as NOEC or ECp) and shall evaluate the intralaboratory variability with a specific reference toxicant for each test method.

2) Ongoing laboratory performance shall be demonstrated by performing regular reference toxicant tests for each test method and species in accordance with the minimum frequency requirements specified in 2.1.a.3.

   i) Intra-laboratory precision on an ongoing basis must be determined through the use of reference toxicant tests and plotted in quality control charts. The control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC50 for acute tests, or as appropriate hypothesis test values, such as the NOEC or NOAEC, over time within a laboratory.

   ii) For endpoints that are point estimates (ICp, ECp) control charts are constructed by plotting the cumulative mean and the control limits which consist of the upper and lower 95% confidence limits (+ 2 std. dev.); these values are re-calculated with each successive test result. For endpoints from hypothesis tests (NOEC, NOAEC) the values are plotted directly and the control limits consist of one concentration interval above and below the concentration representing central tendency (i.e. the mode).

Typographical Correction: The upper and lower 95% confidence limits are defined by $^{+}/_{-}$ 2 standard deviations.

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   iii) After 20 data points are collected for a test method and species, the control chart is maintained using only the 20 most recent data points, i.e. each successive mean value and control limit is calculated using only the last 20 values.

   iv) Control chart limits are expected to be exceeded occasionally regardless of how well a laboratory performs. Acceptance limits for point estimates (ICp, ECp) which are based on 95% confidence limits should theoretically be exceeded for one in twenty tests. Depending on the dilution factor and test sensitivity, control charts based on hypothesis test values (NOEC, NOAEC) may be expected to be exceeded on a similar frequency. Test results which fall outside of control chart limits at a frequency of 5% or less, or which fall just outside control chart limits (especially in the case of highly proficient laboratories which may develop relatively narrow acceptance limits over time), are not rejected *de facto*. Such data are evaluated in comparison with control chart characteristics including the width of the acceptance limits and the degree of departure of the value from acceptance limits.

   v) Laboratories shall develop an acceptance/rejection policy for reference toxicant data which considers test dilution factor, test sensitivity (for hypothesis test values), testing frequency, out–of-control test frequency, relative width of acceptance limits and degree of difference between test results and acceptance limits.

vi) In the case of reference toxicant data which fails to meet acceptance criteria, the results of environmental toxicity tests conducted during the affected period may be suspect and regarded as provisional. In this case the test procedure is examined for defects and the test repeated if necessary, using a different batch of organisms, as soon as possible or the data is qualified.

3) The frequency of reference toxicant testing shall comply with the EPA or state permitting authority requirements. The following minimum frequency shall be met:

i) Each batch of test organisms obtained from an outside source, field collection or from laboratory spawning of field-collected species not amenable to routine laboratory culture (for example, sea urchins and bivalve mollusks) must be evaluated with a reference toxicant test of the same type as the environmental toxicity test within the seven days preceding the test or concurrently with the test.

ii) Test organisms obtained from in-house laboratory cultures must be tested with reference toxicant tests at least once each month for each test method. However, if a given species produced by in-house cultures is used only monthly, or less frequently, a reference toxicant test of the same type must be performed with each environmental toxicity test.

iii) For test methods and species commonly used in the laboratory, but which are tested on a seasonal basis (e.g. sea urchin fertilization tests), reference toxicant tests must be conducted for each month the method is in use.

4) These standards do not currently specify a particular reference toxicant and dilution series however, if the state or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements. All reference toxicant tests conducted for a given test method and species must use the same reference toxicant, test concentrations, dilution water and data analysis methods. A dilution factor of 0.5x or greater shall be used for both acute and chronic tests.

5) The reference toxicant tests shall be conducted following the same procedures as the environmental toxicity tests for which the precision is being evaluated, unless otherwise specified in the test method (for example, 10-day sediment tests employ 96-h water-only reference toxicant tests). The test duration, dilution or control water, feeding, organism age, age range and density, test volumes, renewal frequency, water quality measurements, and the number of test concentrations, replicates and organisms per replicate shall be the same as specified for the environmental toxicity test.

b) Negative Control – Control, Brine Control, Control Sediment, Control Soil or Dilution Water –

1) The standards for the use, type and frequency of testing of negative controls are specified by the test methods and by permit or regulation and shall be followed. A negative control is included with each test.

2) Appropriate additional negative controls shall be included when sample adjustments (for example addition of sodium hydroxide for pH adjustment or thiosulfate for dechlorination) or solvent carriers are used in the test.

3) Test Acceptability Criteria (TAC) - The test acceptability criteria (for example, the whole-effluent chronic *Ceriodaphnia* test requires 80% or greater survival and an average 15 young per female in the controls), as specified in the test method must be achieved for both the reference toxicant and the effluent or environmental sample toxicity test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity tests.

D.2.2 Variability and/or Reproducibility

Intralaboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item D.2.1.a above.

D.2.3 Accuracy

This principle is not applicable to Toxicity Testing.

D.2.4 Test Sensitivity

a) If the Dunnett's procedure is used, the statistical minimum significant difference (SMSD) shall be calculated according to the formula specified by the test method and reported with the test results.

b) Estimate the SMSD for non-normal distribution and or heterogenous variances.

c) Point estimates: (LCp, ICp, or ECp) - Confidence intervals shall be reported as a measure of the precision around the point estimate value.

d) The SMSD shall be calculated and reported for only hypothesis test values, such as the NOEC or NOAEC.

D.2.5 Selection of Appropriate Statistical Analysis Methods

a) If required, methods of data analysis and endpoints are specified by language in the regulation, permit or the test method.

b) Dose Response Curves - When required, the data shall be plotted in the form of a curve relating the dose of the chemical or concentration of sample to cumulative percentage of test organisms demonstrating a response such as death.

D.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in toxicity tests is specified in the test method except the reference standard. All reference standards shall be prepared from chemicals which are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in Section 9.4 above.

Typographical Correction: The above reference should read ~~Appendix D.1.6~~Section 9.2~~,~~ instead of ~~D.1~~9.4.

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c) Only reagent-grade water collected from distillation or deionization units (>17 megohm resistivity) is used to prepare reagents.

D.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit or regulation.

D.2.8 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid loss of cultures due to cross-contamination.

b) Laboratory space must be adequate for the types and numbers of tests performed. The building must provide adequate cooling, heating and illumination for conducting testing and culturing; hot and cold running water must be available for cleaning equipment.

c) Air used for aeration of test solutions, dilution waters and cultures must be free of oil and fumes.

d) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s)) and the name(s) of the taxonomic expert(s) must be kept on file at the laboratory. When organisms are obtained from an outside source the supplier must provide this same information.

e) Instruments used for routine measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, and weight shall be calibrated, and/or standardized per manufacturer's instructions and Section 9.4. Temperature shall be calibrated per Section 9.4.2.1. All measurements and calibrations shall be documented.

Calibration – Chemical and Physical Parameters: Instruments used for routine measurements of chemical and physical parameters, such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, weight, and temperature shall be calibrated and/or standardized per manufacturer's instructions and Section 9.4.2.1. All measurements and calibrations shall be documented.

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f) Test temperature shall be maintained as specified for the test method. Temperature control equipment must be adequate to maintain the required test temperature(s). The average daily temperature of the test solutions must be maintained within 1°C of the selected test temperature, for the duration of the test. The minimum frequency of measurement shall be once per 24 hour period. The test temperature for continuous-flow toxicity tests shall be recorded and monitored continuously.

g) Reagant grade water, prepared by any combination of distillation, reverse osmosis, ion exchange, activated carbon and particle filtration, shall meet the following requirements as verified by monthly measurement: conductivity less than or equal to 0.1 umhos or resistivity greater than or equal to 17 megohm, pH 5.5 to 7.5 S.U. and total residual chlorine non-detectable.

g) The quality of the standard dilution water used for testing or culturing must be sufficient to allow satisfactory survival, growth and reproduction of the test species as demonstrated by routine reference toxicant tests and negative control performance. Water used for culturing and testing shall be analyzed for toxic metals and organics whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified. It is recognized that the analyte lists of some methods manuals may not include all potential toxicants, are based on estimates of chemical toxicity available at the time of publication and may specify detection limits which are not achievable in all matrices. However, for those analytes not listed, or for which the measured concentration or detection limit is greater than the method-specified limit, the laboratory must demonstrate that the analyte at the measured concentration or reported detection limit does not exceed one tenth the expected chronic value for the most sensitive species tested and/or cultured. The expected chronic value is based on professional judgment and the best available scientific data. The "USEPA Ambient Water Quality Criteria Documents" and the EPA AQUIRE data base provide guidance and data on acceptability and toxicity of individual metals and organic compounds.

Test Conditions – Water Quality: Water used for culturing and testing shall, at a minimum, be analyzed **annually** for toxic metals and organics.

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i) For each new batch of food used for culturing and testing, the performance of organisms fed with the new food shall be compared with the performance of organisms with a food of known quality in side-by-side tests. If the food is used for culturing, its suitability is determined using a short-term chronic test that measures the effect of food quality on growth or reproduction of each of the relevant test species in culture, using a minimum of four replicates with each food source. Where applicable, foods used only in chronic toxicity tests are compared with a food of known quality in side-by-side, multi-concentration chronic tests, using the reference toxicant regularly employed in the laboratory QA program. In the case of algae, rotifers or other cultured foods, which are collected as a continuous batch, the quality is assessed, using side-by-side tests as described above, each time new nutrient stocks are prepared, a new starter culture is employed or when a significant change in culture conditions occurs. The laboratory shall have written procedures for the statistical evaluation of food acceptance.

j) Food used to culture organisms used in bioaccumulation tests must be analyzed for the compounds to be measured in the bioaccumulation tests.

Test Conditions – Food Quality: The above requirement also applies to bioconcentration and bioavailability tests.

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k) Test chamber size and test solution volume shall be as specified in the test method. All test chambers used in a test must be identical.

l) Test organisms shall be fed the quantity and type food or nutrients specified in the test method. They shall also be fed at the intervals specified in the test methods.

m) All organisms in a test must be from the same source. Where available certified seeds are used for soil tests.

n) All organisms used in tests, or used as broodstock to produce neonate test organisms (for example cladocerans and larval fish), must appear healthy, show no signs of stress or disease and exhibit acceptable survival (90% or greater) during the 24 hour period immediately preceding use in tests.

o) All materials used for test chambers, culture tanks, tubing, etc. and coming in contact with test samples, solutions, control water, sediment or soil or food must be non-toxic and cleaned as described in the test methods. Materials must not reduce or add to sample toxicity. Appropriate materials for use in toxicity testing and culturing are described in the referenced manuals.

p) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the test methods and shall be documented at least quarterly. For algal and plant tests, the light intensity shall be measured and recorded at the start of each test.

q) At a minimum, during aquatic chronic testing DO and pH shall be measured daily in at least one replicate of each concentration. In static-renewal tests DO must be measured at both the beginning and end of each 24-h exposure period and may be measured in old and new solutions prior to organism transfer, or after organism transfer; pH is measured at the end of each exposure period (i.e. in old solutions).

r) All cultures used for testing shall be maintained as specified in the methods manuals. If test organisms are obtained from an outside source, certification of culture methods and conditions must be provided by the supplier for each lot of organisms used in tests.

s) Age and the age range of the test organisms must be as specified in the test method. Supporting information, such as hatch dates and times, times of brood releases and metrics (for example, chironomid head capsule width) shall be documented.

t) The maximum holding time of effluents (elapsed time from sample collection to first use in a test) shall not exceed 36 hours and the last use of the sample in test renewals shall not exceed 72 hours without the permission of the permitting authority.

u) All samples shall be chilled to 4°C during or immediately after collection (see requirements in section 11.3).

v) Organisms obtained from an outside source must be from the same batch.

w) Chronic tests shall have a minimum of four replicates per treatment.

w) The control population of Ceriodaphnia in chronic effluent or receiving water tests shall contain no more than 20% males.

x) Dissolved oxygen and pH in aquatic tests shall be within acceptable range at test initiation and aeration (minimal) is provided to tests if, and only if, acceptable dissolved oxygen concentrations cannot be otherwise maintained or if specified by the test method.

y) The test soils or sediments must be within the geochemical tolerance range of the test organism.

z) An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each test method). The acceptability of the test shall depend on the experience and professional judgment of the technical employee and the permitting authority.

D.3 MICROBIOLOGY TESTING

These standards apply to laboratories undertaking the examination of materials, products and substances involving microbiological analysis, recovery or testing. The procedures involve the culture media, the test sample and the microbial species being isolated, tested or enumerated.

a) Microbiological testing refers to and includes the detection, isolation, enumeration and identification of microorganisms and their metabolites or confirmation of the absence of growth in materials and media. It includes assays using microorganisms as part of a detection system and their use for ecological testing.

b) These standards are concerned with the quality of test results and not specifically with health and safety measures. In the performance of microbiological testing, laboratories must be aware of and have SOPs that conform with local, State, and national regulatory policies for the safety and health of personnel.

D.3.1 Positive and Negative Controls

a) Negative Controls

The laboratory shall demonstrate that the equipment, media and reagents have not been contaminated through sample handling, preparation or environmental exposure. These controls shall include sterility checks of media, blanks such as filtration blanks, bottle, and buffer blanks.

1) All blanks and uninoculated controls specified by the test method shall be prepared and analyzed at the frequency stated in the method and must include the following controls.

2) Analyze (culture) a known negative control using a non-target organism, as a procedural control of the method for each commercial lot of selective media or batch of media prepared in the lab.

3) Except for self-contained chromofluorogenic methods, a minimum of one uninoculated control shall be prepared and analyzed with each batch of samples. The laboratory shall prepare a series of blanks using the equipment. At least one beginning and ending control shall be prepared, with additional controls inserted after every 10 samples, when the same equipment set is used to prepare multiple samples.

b) Positive Controls

Positive culture controls demonstrate that the medium can support the growth of the test organism, and that the medium produces the specified or expected reaction to the test organism.

1) Each lot of media shall be tested with at least one pure culture of a known positive reaction and, except for self-contained chromofluorogenic methods, shall be included with the sample test batch, each month that the media is used.

2) If routine maintenance culturing is not part of a laboratory's testing and pre-prepared media are routinely used, strict control of the storage conditions and expiration date of media shall be maintained. A positive growth control from a known positive sample shall be run with each lot to ensure that the newly prepared media support growth.

3) If the laboratory has at least one known positive result with an appropriate target organism during the month, a separate positive control is not required.

D.3.2 Test Variability/Reproducibility

a) The laboratory must demonstrate its ability to duplicate the results by analyzing duplicative samples or by performing a positive control in duplicate at least once per month.

~~**Sample Duplicates – Positive Results:** If a sample tests positive, repeated field sampling may be required to fulfill duplication requirements.~~

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b) Participation in, collaborative trials, proficiency testing, or interlaboratory comparisons, either formal or informal, must be done.

D.3.3 Method Evaluation

a) In order to demonstrate the suitability of a test method for its intended purpose, the laboratory shall demonstrate and document that the test method meets acceptance criteria either specified by the method or by the EPA or State program requirements. Acceptance criteria must meet or exceed these requirements and must demonstrate that the test method provides correct/expected results with respect to specified detection capabilities, selectivity, and reproducibility.

   1) Laboratories are required to demonstrate proficiency with the test method prior to first use. This can be achieved by simultaneous, side-by-side analysis by several analysts, or in a one person laboratory with repetitive testing or collaborative testing with another laboratory.

   2) Qualitative microbiological test methods in which the response is expressed in terms of presence/absence, shall be validated by estimating, if possible, the specificity and reproducibility. Differences in matrices must be taken into account when testing different sample types.

   3) The validation of microbiological test methods shall be performed under the same conditions as those for routine sample analysis. This can be achieved by using a combination of naturally contaminated products and spiked products with results that can be statistically analyzed to demonstrate that the test meets its intended purpose.

   4) All validation data shall be recorded and stored at least as long as the test method is in force, or if withdrawn from active use, for at least 5 years past the date of last use.

b) Laboratories shall participate in the Proficiency Test programs (interlaboratory) identified by NELAP (4.2.j or 5.3.4).

D.3.4 Test Performance

All growth and recovery media must be checked to assure that the target organisms respond in an acceptable and predictable manner (see D.3.1.b).

D.3.5 Data Reduction

a) The calculations, data reduction and statistical interpretations specified by each test method shall be followed.

b) If the test method specifies colony counts, such as on membrane filter or plated media then the ability of individual analysts to count colonies accurately shall be verified at least once per month, by having two or more analysts count colonies from the same plate. In a one person laboratory, repetitive counting of the same sample or collaborative testing in another laboratory can be used.

D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared in the laboratory from the different chemical ingredients, from commercial dehydrated powders, or may be purchased ready-to-use.

b) Reagents, commercial dehydrated powders and media shall be used within the shelf-life of the product and shall be documented according to 10.5. The laboratory shall retain all manufacturer-supplied "quality specification statements" which may contain such information as shelf life of the product, storage conditions, sampling regimen/rate, sterility information, including acceptability criteria. Performance checks including the organism used, their culture collection reference, a date of issue of

specification, or statements assuring that the relevant product batch meets the product specifications must be verified.

c) Distilled water, deionized water or reverse-osmosis produced water free from bactericidal and inhibitory substances (e.g., demonstrated with the Water Suitability test) shall be used in the preparation of media, solutions and buffers. The quality of the water shall be monitored for chlorine residual, specific conductance, and heterotrophic bacteria plate count on a monthly frequency (when used) and analyzed for metals yearly and evaluated according to the required method. Records shall be maintained on all activities.

d) Media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer's instructions or the test method.

e) All laboratory media shall be checked to ensure they support the growth of specific microbial cultures. In addition, selective media shall be checked to ensure they suppress the growth of non-target organisms. Media purchased pre-prepared from the manufacturer shall be checked monthly except when the use and maintenance of pure cultures is not part of laboratory procedures. Rather than the commonly used streak method, a quantitative procedure where a known (often low) number of relevant organisms are inoculated into the medium under test and the recovery evaluated must be used.

D.3.7 Selectivity

a) All confirmation/verification tests specified by the test method shall be performed according to method protocols.

b) In order to ensure identity and traceability, laboratories shall use reference cultures of microorganisms obtained from a recognized national collection or an organization recognized by the assessor body.

1) Reference cultures may be revived (if freeze-dried) or transferred from slants and subcultured once to provide reference stocks. Appropriate purity and biochemical checks shall be made with the reference stocks and documented. The reference stocks shall be preserved by a technique which maintains the characteristics of the strains. Examples of such methods are freeze-drying, liquid nitrogen storage and deep-freezing. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they must not be re-frozen and re-used.

2) Working stocks shall not be sequentially cultured more than five times except when:

i. It is required by standard test methods, or

ii. Laboratories can provide documentary evidence demonstrating that there has been no loss of viability, no changes in biochemical activity and/or no change in morphology.

3) Working stocks shall not be subcultured to replace reference stocks.

4) A scheme for handling reference cultures is included in Figure D-1.

5) Where used, a new reference culture must be obtained on at least an annual basis.

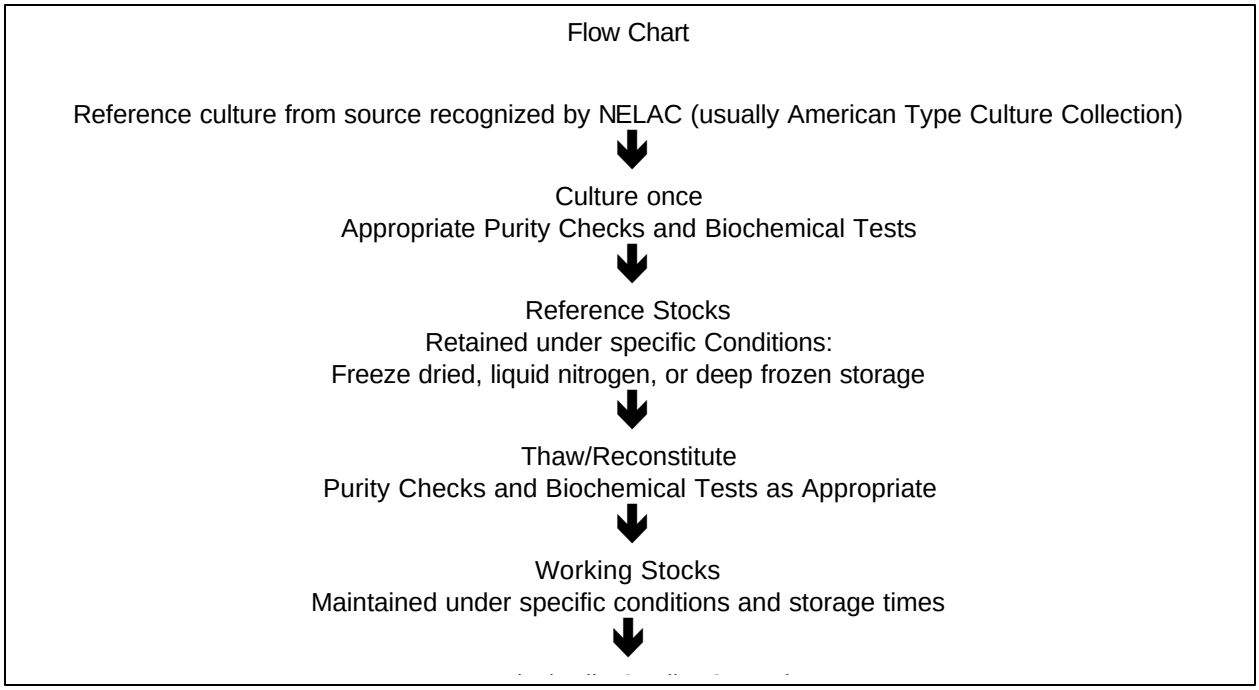

FIGURE D-1. USE OF REFERENCE CULTURES (BACTERIA)

D.3.8 Constant and Consistent Test Conditions

a) The laboratory shall devise an appropriate environmental monitoring program and examine trends in levels of contamination. Acceptable background counts shall be determined and there shall be documented procedures to deal with situations in which these limits are exceeded.

b) Walls, floors, ceilings and work surfaces shall be non-absorbent and easy to clean and disinfect. Wooden surfaces of fixtures and fittings shall be adequately sealed. Measures shall be taken to avoid accumulation of dust by the provision of sufficient storage space, by having minimal paperwork in the laboratory and by prohibiting plants and personal possessions from the laboratory work area.

c) Temperature measurement devices

1) Where the accuracy of temperature measurement has a direct effect on the result of the analysis, temperature measuring devices such as liquid-in-glass thermometers, thermocouple, platinum resistance thermometers used in incubators, autoclaves and other equipment shall be the appropriate quality to meet specification(s) in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement and they shall be calibrated to national or international standards for temperature (see 9.2). Calibration shall be done at least annually.

~~Typographical Correction:~~ ~~The reference at the end of this paragraph should read Section 9.2 instead of Section 9.2.1.~~

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2) Demonstration of sterilization shall be provided by a continuous temperature recording and through the use of appropriate biological indicators at least once each month of use except when

temperature recording is not available and then the frequency of biological indicator use shall be once each week.

3) The stability and uniformity of temperature distribution and time after test sample addition to re-establish equilibrium conditions in incubators, water baths, ovens and temperature controlled rooms shall be established.

d) Autoclaves

1) The performance of each autoclave shall be initially evaluated by establishing its functional properties and performance, for example heat distribution characteristics with respect to typical uses. Autoclaves shall meet specified temperature tolerances. Pressure cookers fitted only with a pressure gauge are not recommended for sterilization of media or decontamination of wastes.

2) Records of autoclave operations including temperature and time shall be maintained. This shall be done for every cycle. Acceptance/rejection criteria shall be established and used to evaluate the autoclave efficiency and effectiveness.

e) Volumetric equipment such as automatic dispensers, dispenser/diluters, mechanical hand pipettes and disposal pipettes used in the microbiology laboratory shall be calibrated, as outlined in Section 9.4.2.1 and documented. Each lot of disposable pipets requires a manufacturer's verification of accuracy and these records shall be retained.

f) UV Instruments

1) Are to be tested quarterly for effectiveness by testing of the power output of the UV bulb in the UV instruments.

g) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements (see Section 9.4). Mechanical timers shall be checked regularly against electronic timing devices to ensure accuracy.

h) Glassware

1) Glassware shall be tested for possible presence of residues which may inhibit or promote growth of microorganisms by performing the Inhibitory Residue Test each time the lab changes the lot of detergent, personnel, or washing procedures.

2) Each batch of washed glassware shall be tested for possible acid or alkaline residue by testing one piece of glassware with a suitable pH indicator such as bromthymol blue.

D.4 RADIOCHEMICAL TESTING

These standards apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. These procedures for radiochemical analysis may involve some form of chemical separation followed by detection of the radioactive decay of analyte (or indicative daughters) and tracer isotopes where used. For the purpose of these standards procedures for the determination of radioactive isotopes by mass spectrometry (e.g. ICP-MS or TIMS) or optical (e.g. KPA) techniques are not addressed herein.

D.4.1 Negative and Positive Controls

a) Negative Controls

1) Method Blank - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The method blank result shall be assessed against the specific acceptance criteria [see 10.1.2.b)18] specified in the laboratory method manual [see 10.1.2]. When the specified method blank acceptance criteria is not met the specified corrective action and contingencies [see 10.1.2.b)19 and 20] shall be followed and results reported with appropriate data qualifying codes. The occurrence of a failed method blank acceptance criteria and the actions taken shall be noted in the laboratory report [Section 13.a)10].

2) In the case of gamma spectrometry where the sample matrix is simply aliquoted into a calibrated counting geometry the method blank shall be of similar counting geometry that is empty or filled to similar volume with ASTM Type II water to partially simulate gamma attenuation due to a sample matrix.

3) There shall be no subtraction of the required method blank [see D.4.1.a)1] result from the sample results in the associated preparation or analytical batch unless permitted by method or program. This does not preclude the application of any correction factor (e.g., instrument background, analyte presence in tracer, reagent impurities, peak overlap, calibration blank, etc.) to all analyzed samples, both program/project submitted and internal quality control samples. However, these correction factors shall not depend on the required method blank result in the associated analytical batch.

4) The method blank sample shall be prepared with similar aliquot size to that of the routine samples for analysis and the method blank result and acceptance criteria [10.1.2.b)18] shall be calculated in a manner that compensates for sample results based upon differing aliquot size.

b) Positive Controls

Typographical Correction: ~~The references to Section 10.1.2.a)19 and 20 in Section D.4.2, D.4.3, and D.4.4 should read Section 10.1.2.**b**)19 and 20.~~

~~**D-20**~~

1) Laboratory Control Samples - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The laboratory control sample result shall be assessed against the specific acceptance criteria [see 10.1.2.b)18] specified in the laboratory method manual [see 10.1.2]. When the specified laboratory control sample acceptance criteria is not met the specified corrective action and contingencies [see 10.1.2.b)19 and 20] shall be followed. The occurrence of a failed laboratory control sample acceptance criteria and the actions taken shall be noted in the laboratory report [see 13.a)10.]

2) Matrix Spike - Shall be performed at a frequency of one per preparation batch for those methods which do not utilize an internal standard or carrier, for which there is a chemical separation process, and where there is sufficient sample to do so. The exceptions are gross alpha, gross beta and tritium which shall require matrix spikes for aqueous samples. The results of this analysis shall be one of the quality control measures to be used to assess the batch. The matrix spike result shall be assessed against the specific acceptance criteria [see 10.1.2.b)18] specified in the laboratory method manual [see 10.1.2]. When the specified matrix spike acceptance criteria is not met, the specified corrective action and contingencies [see 10.1.2.b)19 and 20] shall be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report [see 13.a)10]. The lack of sufficient sample aliquot size to perform a matrix spike shall be noted in the laboratory report.

3) The activity of the laboratory control sample shall: (1) be two to ten times the detection limit or (2) at a level comparable to that of routine samples if the sample activities are expected to exceed 10 times the detection limit.

4) The activity of the matrix spike analyte(s) shall be greater than ten times the detection limit.

5) The laboratory standards used to prepare the laboratory control sample and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration.

6) The matrix spike shall be prepared by adding a known activity of target analyte. Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g.– plutonium, Pu 238 and Pu 239, using alpha spectrometry), only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified detection limit each shall be assessed against the specified acceptance criteria.

7) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope, the laboratory control sample and matrix spike shall contain isotopes that represent the low (e.g. americium-241), medium (e.g. cesium-137) and high (e.g. cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

8) The laboratory control sample shall be prepared with similar aliquot size to that of the routine samples for analyses.

c) Other Controls

1) Tracer – For those methods that utilize a tracer (i.e. internal standard) each sample result shall have an associated tracer recovery calculated and reported. The tracer recovery for each sample result shall be one of the quality control measures to be used to assess the associated sample result acceptance. The tracer recovery shall be assessed against the specific acceptance criteria [see 10.1.2.b)18] specified in the laboratory method manual [see 10.1.2]. When the specified tracer recovery acceptance criteria is not met the specified corrective action and contingencies [see 10.1.2.b)19 and 20] shall be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 13.a)10].

2) Carrier – For those methods that utilize a carrier, each sample shall have an associated carrier recovery calculated and reported. The carrier recovery for each sample shall be one of the quality control measures to be used to assess the associated sample result acceptance. The carrier recovery shall be assessed against the specific acceptance criteria [see 10.1.2.b)18] specified in the laboratory method manual [see 10.1.2]. When the specified carrier recovery acceptance criteria is not met the specified corrective action and contingencies [see 10.1.2.b)19 and 20] shall be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 13.a)10].

D.4.2 Analytical Variability/Reproducibility

a) Replicate - Shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria [see 10.1.2.b)18] specified in the laboratory method manual [see 10.1.2]. When the specified replicate acceptance criteria is not met the specified corrective action and contingencies [see 10.1.2.b)19 and 20] shall be followed. The corrective action shall consider the fact that sample inhomogeneity may be a cause of the failed replicate acceptance criteria. The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report [see 13.a)10].

b) For low level samples (less than approximately three times the detection limit) the laboratory may analyze duplicate laboratory control samples or a replicate matrix spike (matrix spike and a matrix spike duplicate) to determine reproducibility within a preparation batch.

D.4.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Initial Demonstration of Capability - (section 10.2.1 and Appendix C) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel or method.

b) Proficiency Test Samples - The results of such analysis (4.2.j and 5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.4.4 Radiation Measurement System Calibration

~~**Typographical Correction:** The reference in Sections D.4.6.a) and D.4.6.g) to Section 10.1.2.13 should read Section 10.1.2.**b**)13.~~

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Because of the stability and response nature of modern radiation measurement instrumentation, it is not typically necessary to verify calibration of these systems each day of use. This section addresses those practices that are necessary for proper calibration and those requirements of section 9.4.2 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

a) Initial Instrument Calibration

1) Given that activity detection efficiency is independent of sample activity at all but extreme activity levels, the requirements of subsections f, h and i of 9.4.2.1 are not applicable to radiochemical method calibrations except mass attenuation in gas-proportional counting and sample quench in liquid scintillation counting. Radiochemistry analytical instruments are subject to calibration when purchased, when the instrument is serviced, when the instrument is moved and when the instrument setting(s) have been changed.

2) Instrument calibration shall be performed with reference standards as defined in section D.4.7a. The standards shall have the same general characteristics (i.e., geometry, homogeneity, density, etc.) as the associated samples.

3) The frequency of calibration shall be addressed in the laboratory method manual [see 10.1.2.b)13] if not addressed in the method. A specific frequency (e.g. monthly) or observations from the associated control or tolerance chart, as the basis of calibration shall be specified.

b) Continuing Instrument Calibration Verification

Calibration verification checks shall be performed using appropriate check sources and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the calibration has not changed. The same check source used in the preparation of the tolerance chart or control chart at the time of calibration shall be used in the calibration verification of the instrument. The check sources must provide adequate counting statistics for a relatively short count time and the source should be sealed or encapsulated to prevent loss of activity and contamination of the instrument and laboratory personnel. For alpha and gamma spectroscopy systems, the instrument calibration verification shall include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.

1) For gamma spectroscopy systems, the calibration verification checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.

2) For alpha spectroscopy systems, the calibration verification check for energy calibration shall be performed on a weekly basis and the performance check for counting efficiency shall be performed on at least a monthly basis.

3) For gas-proportional and liquid scintillation counters, the calibration verification check for counting efficiency shall be performed on a day of use basis. Verification of instrument calibration does not directly verify secondary calibrations, e.g., the mass efficiency curve or the quench curve.

4) For scintillation counters the calibration verification for counting efficiency shall be performed on a day of use basis.

c) Background Measurement

Background measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory maintains its capability to meet required data quality objectives. These values are subtracted from the total measured activity in the determination of the sample activity.

1) For gamma spectroscopy systems, background measurements shall be performed on at least a monthly basis.

2) For alpha spectroscopy systems, background measurements shall be performed on at least a monthly basis.

3) For gas-proportional counters background measurements shall be performed on a weekly basis.

4) For scintillation counters, background measurements shall be performed each day of use.

D.4.5 Detection Limits

a) Must be determined prior to sample analysis and must be redetermined each time there is a significant change in the test method or instrument type.

b) The procedures employed must be documented and consistent with mandated method or regulation.

D.4.6 Data Reduction

a) Refer to Section 10.6, “Computers and Electronic Data Related Requirements,” of this document.

b) Measurement Uncertainties – Each result shall be reported with the associated measurement uncertainty. The procedures for determining the measurement uncertainty must be documented and be consistent with mandated method and regulation.

D.4.7 Quality of Standards and Reagents

a) The quality control program shall establish and maintain provisions for radionuclide standards.

1) Reference standards that are used in a radiochemical laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. Any reference standards purchased outside the United

States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards shall conform to ANSI N42.22 to assure the quality of their products.

2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.

3) Laboratories should consult with the supplier if the lab's verification of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory shall not use a value other than the decay corrected certified value.

b) All reagents used shall be analytical reagent grade or better.

D.4.8 Constant and Consistent Test Conditions

a) To prevent incorrect analysis results caused by the spread of contamination among samples, the laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

b) For gamma spectroscopy systems, background check measurements shall be performed each day of use.

c) For alpha spectroscopy systems, background check measurements shall be performed except when using the electro-plating method of sample preparation.

d) For gas-proportional counter systems, background check measurements shall be performed each day of use.

D.5 AIR TESTING

These standards shall apply to samples that are submitted to a laboratory for the purpose of analysis. They do not apply to field activities such as source air emission measurements or the use of continuous analysis devices.

D.5.1 Negative and Positive Controls

a) Negative Controls

1) Method Blanks – Shall be performed at a frequency of at least one (1) per batch of twenty (20) environmental samples or less per sample preparation method. The results of the method blank analysis shall be used to evaluate the contribution of the laboratory provided sampling media and analytical sample preparation procedures to the amount of analyte found in each sample. If the method blank result is greater than the detection limit and contributes greater than 10% of the total amount of analyte found in the sample, the source of the contamination must be investigated and measures taken to eliminate the source of contamination. If contamination is found, the data shall be qualified in the report.

2) Collection Efficiency – Sampling trains consisting of one or more multi-section sorbent tube, that are received intact by the laboratory, shall be separated into “front” and “back” sections if required by the client. Each section shall be processed and analyzed separately and the analytical results reported separately.

b) Positive Controls

1) Laboratory Control Sample (LCS) – Shall be analyzed at a rate of at least one (1) per batch of twenty (20) or fewer samples per sample preparation method for each analyte. If a spiking solution is not available, a calibration solution whose concentration approximates that of the samples, shall be included in each batch and with each lot of media. The concentration of the LCS shall be relevant to the intended use of the data and either at a regulatory limit or below it.

c) Surrogates – Shall be used as required by the test method.

d) Matrix Spike – Shall be used as required by the test method.

D.5.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates – Shall be analyzed at a minimum of 1 in 20 samples per sample batch. The laboratory shall document their procedure to select the use of appropriate types of spikes and duplicates. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the spikes and duplicates may indicate a problem with the sample composition and shall be reported to the client.

D.5.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) Demonstration of Capability – (Sections 6.2 and 10.2.1) shall be performed prior to the analysis of any samples and with a significant change in instrument type, personnel, matrix, or test method.

b) Calibration – Calibration protocols specified in Section 9.4 shall be followed.

c) Proficiency Test Samples – The results of such analyses (4.2.j or 5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.5.4 Detection Limits

The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the mandated test method or applicable regulation, e.g., MDL. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

a) A detection limit study is not required for any component for which spiking solutions are not available such as temperature or on-line analyses.

b) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).

c) Detection limits must be determined each time there is a significant change in the test method or instrument type.

d) All sample processing steps of the analytical method must be included in the determination of the detection limit.

e) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.

f) The laboratory must have established procedures to tie detection limits with quantitation limits.

D.5.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.5.6 Quality of Standards and Reagents

a) The source of standards shall comply with 9.2.

b) The purity of each analyte standard and each reagent shall be documented by the laboratory through certificates of analyses from the manufacturer/vendor, manufacturer/vendor specifications, and/or independent analysis.

Expiration Date of Standards and Reagents: The date of expiration of each analyte standard and each reagent shall be documented by the laboratory, in addition to the purity.

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c) In methods where the purity of reagents is not specified, analytical reagent grade or higher quality, if available, shall be used.

D.5.7 Selectivity

The laboratory shall develop and document acceptance criteria for test method selectivity such as absolute and relative retention times, wavelength assignments, mass spectral library quality of match, and mass spectral tuning.

D.5.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) The laboratory shall document that all sampling equipment, containers and media used or supplied by the laboratory meet required test method criteria.

c) If supplied or used by the laboratory, procedures for field equipment decontamination shall be developed and their use documented.

d) The laboratory shall have a documented program for the calibration and verification of sampling equipment such as pumps, meter boxes, critical orifices, flow measurement devices and continuous analyzers, if these equipment are used or supplied by the laboratory.

DoD APPENDICES

APPENDIX DOD-A – REPORTING REQUIREMENTS

The reporting requirements outlined below are for hard-copy data reports from the laboratory. They are divided into mandatory requirements for all printed data reports, and optional requirements. Optional reporting requirements are those that may be required by a specific project, depending upon the needs of the project. The following elements are required in every report: cover sheet, table of contents, case narrative, analytical results, sample management records, and QA/QC information. Information for third-party review and a performance-based data package may be required depending on project-specific requirements or the method being used.

1. Cover Sheet. The cover sheet shall specify the following information:

- title of report (i.e., Test Report, Test Certificate)
- name and location of laboratory (to include a point of contact, phone and facsimile numbers)
- name and location of any subcontractor laboratories, and appropriate test method performed
- contract number
- client name and address
- project name and site location
- statement of data authenticity and official signature and title of person authorizing report release
- amendments to previously released reports that clearly identify the serial number for the previous report and state the reason(s) for reissuance of the report.

2. Table of Contents. Laboratory data packages should be organized in a format that allows for easy identification and retrieval of information. An index or table of contents shall be included for this purpose.

3. Case Narrative. A case narrative shall be included in each report. The purpose of the case narrative is to:

- describe any abnormalities and deviations that may affect the analytical results, and
- summarize any issues in the data package that need to be highlighted for the data user to help them assess the usability of the data.

The case narrative shall provide:

- a table(s) summarizing samples received, providing a correlation between field sample numbers and laboratory sample numbers, and identifying which analytical test methods were performed and by which laboratories
- a list of samples that were received but not analyzed
- a description of extractions or analyses that are performed out of holding times
- a definition of all data qualifiers or flags used
- identification of deviations of any calibration standards or QC sample results from appropriate acceptance limits and a discussion of the associated corrective actions taken by the laboratory
- appropriate notation of any other factors that could affect the sample results (e.g., air bubbles in VOC sample vials, excess headspace in soil VOC containers, the presence of multiple phases, sample temperature and sample pH excursions, container type or volume, etc.).

4. Analytical Results. The results for each sample shall contain the following information at a minimum: (Information need not be repeated if noted elsewhere in the data package.)

- project name and site location
- field sample ID number as written on custody form
- laboratory sample ID number
- matrix (soil, water, oil, etc.)
- date sample extracted or prepared

- date sample analyzed
- method numbers for all preparation, cleanup, and analysis procedures employed
- analyte or parameter
- method reporting limits and method quantitation limits (at or above the low-level standard concentration) adjusted for sample-specific factors (e.g., aliquot size, dilution/concentration factors, moisture content)
- method detection limits
- analytical results with correct number of significant figures
- any data qualifiers assigned
- concentration units
- dilution factors
- any dilutions or concentrations for all reported data, and if neat or less diluted results are available, recorded and reported data from both runs
- percent moisture or percent solids (all soils are to be reported on a dry weight basis).

The following information is optional but may be required site specifically:

- laboratory name and location (city and state)
- sample description
- sample preservation or condition at receipt
- date sample collected
- date sample received
- sample aliquot analyzed
- final extract volume
- CAS numbers.

5. Sample Management Records. These types of records include the documentation accompanying the samples:

- chain-of-custody records
- shipping documents
- records generated by the laboratory which detail the condition of the samples upon receipt at the laboratory (e.g., sample cooler receipt forms)
- telephone conversation records associated with actions taken or quality issues
- laboratory internal sample custody records through sample analysis, transfer, and disposal.

6. QA/QC Information. The minimum internal QC data package must include:

- matrix spikes percent recovery
- relative percent difference (RPD) of required duplicates
- LCS percent recoveries
- surrogate percent recoveries (organics)
- tracer recoveries (radiochemical)
- method blank results
- preparation, analysis, and other batch numbers.

7. Information for Third-Party Review. The information listed below is required if third-party (from outside the laboratory) data validation or verification is to be performed. This information is therefore optional and is provided only when the project-specific requirements specify that a third-party review will occur:

- calibration data from the initial calibration curve
- initial calibration verification (ICV)
- continuing calibration verification(s) (CCV)
- performance standards analyzed in conjunction with the test method (e.g., tuning standards, degradation check standards, etc.)

- preparation, analysis, and other batch numbers[1]
- raw data (e.g., chromatograms, mass spectrum results)
- matrix spike (MS), if applicable (includes spike target concentration levels, measured spike concentration, and calculated recoveries)[1]
- RPD of required duplicates (e.g., MS, LCS, field duplicates)[1]
- method blank results[1]
- LCS recoveries[1]
- surrogate recoveries (organics)[1]
- serial dilutions (SD) percent difference (inorganics)
- post-digestion spikes recovery (inorganics).

In addition, the data package for third party review may include:

- method detection limit studies and
- supporting documentation (e.g., run logs, sample preparation logs, standard preparation logs).

The data validation guidelines for performance-based methods established in other DoD guidance on data review and data validation, EPA national functional guidelines, EPA regional functional guidelines, and project-specific guidelines for validation may all have distinct reporting formats. The appropriate validation guidelines should be consulted to determine what type of data package is required.

8. Performance Based Data Package. The requirements for the ***performance based data package are the same as those defined within the definitive data package with the addition of the following items: (1) all appropriate project action level(s) and DQOs, and (2) appropriate preparatory and analysis logs.*** Refer to other DoD guidance on the data review of performance based methods for further details on this data package.

[1] Required for other purposes identified in number 6, QA/QC Information.

APPENDIX DOD-B – QUALITY CONTROL REQUIREMENTS

The quality control (QC) protocols specified by the method shall be followed. In some cases the method may be ambiguous or provide insufficient detail. The specific manner in which methods commonly used by DoD should be implemented is detailed in the following tables. Modifications to the following requirements need project-specific approval by DoD personnel.

The tables describe specific quality assurance and quality control requirements for analytical methods (SW-846) commonly used when investigating DoD sites. The tables specify the method requirements, when available, as well as additional clarification and/or requirements from DoD. If possible, the actual requirement from the method is listed, although in some cases the description in the method is so lengthy that only a reference to the appropriate section is made. The methods should always be referenced, however, for clarification purposes. DoD has done its best to interpret the methods, providing clarification where there are inconsistencies between existing guidance documents, and stating DoD preferences when multiple options are acceptable. If there is a contradiction between the method and the following tables, the requirements specified in the tables shall be followed.

SW-846 Methods

This appendix refers to the method versions current at the time of publication. As methods are updated subsequent versions of this manual will incorporate the changes. If the tables in this appendix do not yet correspond with the most recent version of the SW-846 method, or a new method that analyzes for the same group of analytes becomes available, the requirements in the tables shall be followed where appropriate. Otherwise, follow the requirements in the method.

Table B-1 below presents a summary of the definition, purpose, and evaluation of the major QC checks required in the subsequent QA/QC tables (B-2 through B-10) for the various methods. The *definition* column describes generally what the QC check is and/or how it is performed. The *purpose* column describes why the check is important for assessing and measuring the quality of the data being generated. The *evaluation* column describes how to interpret the results of the QC check, particularly in the context of the results of other QC checks. This table should be used in conjunction with the instrument- and method-specific requirement tables to properly implement the methods for DoD projects. In addition, a supplementary list of acronyms and a glossary relevant to this appendix follows Table B-10.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION

QC Check	Definition	Purpose	Evaluation
Breakdown check (Endrin - Method 8081A only, DDT - Methods 8081A and 8270C)	Analysis of a standard solution containing Endrin and DDT. Area counts of these compounds and their breakdown products are evaluated to assess instrument conditions.	To verify the inertness of the injection port because DDT and Endrin are easily degraded in the injection port.	If degradation of either DDT or Endrin exceeds method-specified criteria, corrective action must be taken before proceeding with calibration.
Calibration blank (metals only)	Reagent water containing no analytes of interest, but acidified to the same pH as all samples.	To determine the zero point of the calibration curve for all initial and continuing calibrations.	
Confirmation of positive results (organics only)	Use of alternative analytical techniques (another method, dissimilar column, or different detector) to validate the presence of target analytes identified.	To verify the identification of an analyte.	This is a required QC procedure. All positive results must be confirmed.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (Continued)

QC Check	Definition	Purpose	Evaluation
Continuing calibration verification (CCV)	The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. (IDQTF)	To verify that instrument response is reliable, and has not changed significantly from the current initial calibration curve.	If the values for the analytes are outside the acceptance criteria, the initial calibration may not be stable. Results associated with out-of-control CCV results require reanalysis or flagging.
Demonstrate acceptable analyst capability	Analyst runs QC samples in series to establish his ability to produce data of acceptable accuracy and precision.	To establish the analysts' ability to produce data of acceptable accuracy and precision.	The average recovery and standard deviation of the replicates must be within designated acceptance criteria. Analysis of field samples cannot be conducted until this check is successful.
Dilution test (metals only)	Analysis of a positive sample, which has been diluted to a concentration five times the original, to confirm that there is no interference at lower concentrations. (Modified COE)	To assess matrix interference.	Agreement within 10% between the concentration for the undiluted sample and five times the concentration for the diluted sample indicates the absence of interferences, and such samples may be analyzed without using the method of standard additions. Results outside acceptance limits indicate a possible matrix effect. For ICP, a post-digestion spike must be run; for GFAA, a recovery test must be run.
Distilled standards (one high and one low) (cyanide only)	Standards are run through the distillation procedure and then compared to the undistilled standards' reported values. (Method)	To check the efficiency of the distillation process.	Results must agree to within ±10% of the undistilled value before analysis can proceed.
Duplicate sample	Two identical portions of material collected for chemical analysis, and identified by unique alphanumeric codes. The duplicate may be portioned from the same sample, or may be two identical samples taken from the same site. The two portions are prepared and analyzed identically. (Modified QSM)	To provide information on the heterogeneity of the sample matrix or to determine the precision of the intralaboratory analytical process for a specific sample matrix.	A duplicate sample will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the relative percent difference between the sample and the sample duplicate. If the sample matrix is homogeneous (such as with drinking water) and the relative percent difference is high, this could indicate a problem in the analytical system.
GC column performance check (Methods 8280A and 8290 only)	Analysis of method-specified compounds to verify chromatographic separation of dioxin isomers. (Method)	To evaluate the performance of the analytical system and establish retention time window markers for dioxin isomers.	Sample analysis cannot begin until method-specified criteria are met.
Initial calibration for all analytes (ICAL)	Analysis of analytical standards at different concentrations that are used to define the linearity and dynamic range of the response of the analytical detector or method. (Guide to Environmental Analytical Methods, 2nd Edition)	To establish a calibration curve for the quantification of the analytes of interest.	Statistical procedures are used to determine the relationship between the signal response and the known concentration of analytes of interest. The initial calibration must be successful before any samples or other QC check samples can be analyzed.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (Continued)

QC Check	Definition	Purpose	Evaluation
Instrument detection limit (IDL) study (ICP/MS Method 6020 only)	The process to determine the minimum concentration of a substance (analyte) that an instrument can differentiate from noise. The procedure for calculating varies by method.	To provide a quarterly evaluation of instrument sensitivity.	IDLs must be established quarterly for Method 6020.
Interference check solutions (ICP Only)	A pair of solutions containing interfering elements that are used to verify the correction factors of analytes of concern.	To verify the established correction factors by analyzing the interference check solution at the beginning of the analytical sequence.	No samples can be run if this check does not pass acceptance criteria.
Internal standards	A known amount of standard added to all standards and samples as a reference for evaluating and controlling the precision and bias of the applied analytical method. (QSM)	To verify that the analytical system is in control.	Any samples associated with out-of-control results must be reanalyzed.
Laboratory control sample (LCS) containing all reported analytes	A QC standard of known composition prepared using reagent free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is analyzed using the same sample preparation, reagents, and analytical methods employed for regular samples. (Guide to Environmental Analytical Methods, 2nd Edition)	To evaluate method performance by assessing the ability of the laboratory/analyst to successfully recover the target analytes from a control (clean) matrix. Control limits for LCS recovery, typically expressed as percent recovery, are used for the development of statistical control limits and serve as acceptance criteria for determining whether an analytical run is in control (batch acceptance).	This is a required QC check. The inability to achieve acceptable recoveries in the LCS indicates problems with the accuracy/bias of the measurement system. Failure to achieve acceptable recoveries in a "clean" matrix is an indicator of possible problems achieving acceptable recoveries in field samples.
Linear range or high-level calibration check standard (ICP only)	High-level calibration check standard periodically analyzed to verify the linearity of the calibration curve at the upper end.	To verify quantitative accuracy of data up to the high-level concentration.	This QC check establishes the upper linear range of the calibration.
Low-level calibration check standard (ICP only)	A reference standard that contains a small quantity of analyte (less than or equal to the quantitation limit).	To confirm the accuracy of measurements at or near the quantitation limit. It establishes the lower quantitation limit of the calibration curve for those ICP methods that rely on single point calibration. It also may be used to validate a client's reporting limit.	This QC check must be within acceptance criteria before any samples are analyzed.
Matrix spike	A sample prepared by adding a known concentration of a target analyte to an aliquot of a specific environmental sample for which an independent estimate of the target analyte concentration is available. (Modified G-5)	To assess the performance of the method as applied to a particular project matrix. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. The recovery of target analytes from the matrix spike sample is used to determine the bias of the method in the specific sample matrix.	The lack of acceptable recoveries in the matrix spike often points to problems with the sample matrix. One test of this is a comparison to the LCS recoveries. If the corresponding LCS recoveries are within acceptable limits, a matrix effect is likely. The laboratory should not correct for recovery; only report the results of the analyses and the associated matrix spike results and indicate that the results from these analyses have increased uncertainty.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (Continued)

QC Check	Definition	Purpose	Evaluation
Matrix spike duplicate (MSD)	A second replicate matrix spike prepared in the laboratory, spiked with identical, known concentrations of targeted analyte(s), and analyzed to obtain a measure of the precision of the recovery for each analyte. (Modified QSM)	To assess the performance of the method as applied to a particular project matrix and provide information on the homogeneity of the matrix. Used to determine the precision of the intralaboratory analytical process for a specific sample matrix.	When compared to the MS, the MSD will provide information on the heterogeneity of the sample matrix. The greater the heterogeneity of the matrix, the greater the RPD between the matrix spike and the matrix spike duplicate. Also, if the sample matrix is more homogeneous, such as with drinking water, and the RPD is high, this could indicate a problem in the analytical system.
Matrix verification sample (hexavalent chromium only)	A pH-adjusted filtrate that has been spiked with hexavalent chromium to ensure that the sample matrix does not have a reducing condition or other interferents that could affect color development. (Modified Method)	To ensure that the sample matrix does not have a reducing condition or other interferents that affect color development.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. If the result of verification indicates a suppressive interference, the sample should be diluted and reanalyzed. If the interference persists after sample dilution, an alternative method (Method 7195, Coprecipitation, or Method 7197, Chelation/Extraction) should be used.
Method detection limit (MDL) verification check	A low-level spike taken through the preparatory and analytical steps at two times the MDL used to verify that the lab can detect analytes at the calculated MDL.	To validate the MDL on an ongoing basis.	If the MDL verification check fails, the MDL verification check shall be reprepped and reanalyzed at a higher level to set a higher MDL or the MDL study must be repeated.
Method blank	A sample of a matrix similar to the batch of associated samples (when available) in which no target analytes or interferences are present at concentrations that impact the analytical results. It is processed simultaneously with samples of similar matrix and under the same conditions as the samples. (Modified QSM)	To assess background interference or contamination that exists in the analytical system that might lead to the reporting of elevated concentration levels or false positive data. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of bias introduced by the preparation and analytical procedure.	This is one of the QC samples used to measure laboratory accuracy/bias. This sample could indicate whether contamination is occurring during sample preparation and analysis. If analytes are detected $\geq$ ½RL, reanalyze or qualify (B-flag) all results for the specific analyte(s) in all samples in the associated preparatory batch, as appropriate.
MDL study **(Also known as an IDL study in SW-846 Method 6020, ICP/MS)**	The process to determine the minimum concentration of a substance (analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40CFR part 136 Appendix B)	To determine the lowest concentration of an analyte that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero.	MDLs must be established prior to sample analysis. The reporting or quantitation limit is at least three times the MDL. Used in combination with the MDL verification check to validate the MDL on an ongoing basis.
Method of standard additions (ICP/GFAA only)	Adding known amounts of standard to one or more aliquots of the processed sample solution. (Method)	To compensate for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift.	This is the method used when matrix interferences are present and do not allow determination of accurate sample results.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (Continued)

QC Check	Definition	Purpose	Evaluation
Post digestion spike addition (ICP only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects.	To confirm the presence of a matrix interference. Assess matrix effects based on, (1) the occurrence of new and unusual matrices included within the batch, or (2) contingency analysis based on serial dilution or matrix spike failures.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. Results outside the acceptance limits require a method of standard additions (MSA) for all samples within the batch.
Recovery test (GFAA only)	An analyte spike added to a portion of prepared sample to verify absence or presence of matrix effects.	To confirm the presence of a matrix interference. Assess matrix effects based on, (1) the occurrence of new and unusual matrices included within the batch, or (2) contingency analysis based on serial dilution or matrix spike failures.	To verify the absence of an interference, the spike recovery must be between 85% and 115%. Results outside the acceptance limits require a MSA for all samples within the batch.
Retention time window position establishment for each analyte (and surrogate) (all chromatographic methods only)	Determination of the placement of the retention time window (i.e., start/stop time) of each analyte or group of analytes as it elutes through the chromatographic column so that analyte identification can be made during sample analysis. This is done during the initial calibration.	To identify analytes of interest.	Incorrect window position may result in false negatives, require additional manual integrations, and/or cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified.
Retention time window verification for each analyte (and surrogate) (all chromatographic methods only)	A standard is used to verify that the width and position of the retention time windows are valid so that accurate analyte identification can be made during sample analysis.	To minimize the occurrence of both false positive and false negative results at each calibration verification.	The peaks from the standard used are compared to the retention time window established during the initial calibration (ICAL) to verify that the analytes of interest still fall within the window.
Retention time window width calculated for each analyte (and surrogate) (non-MS chromatographic methods only)	Determination of the length of time between sample injection and the appearance of a peak at the detector. The total length of time (window) is established for each analyte or groups of analytes and is set for complete elution of analyte peaks. It is based upon a series of analyses and statistical calculations that establish the measured band on the chromatogram that can be associated with a specific analyte or group of analytes.	To ensure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the standards and sample matrix to be analyzed. It is done to minimize the occurrence of both false positive and false negative results.	Used to evaluate continued system performance. Tight retention time windows may result in false negatives and/or may cause unnecessary reanalysis of samples when surrogates or spiked compounds are erroneously not identified. Overly wide retention time windows may result in false positive results that cannot be confirmed upon further analysis.
Second source calibration verification	A standard obtained or prepared from a source independent of the source of standards for the initial calibration. Its concentration should be at or near the middle of the calibration range. It is done after the initial calibration. (QSM)	To verify the accuracy of the initial calibration.	The concentration of the second-source calibration verification, determined from the analysis, is compared to the known value of the standard to determine the accuracy of the ICAL. This independent verification of the ICAL must be acceptable before sample analysis can begin.

TABLE B-1. SUMMARY OF QUALITY CONTROL CHECK DEFINITIONS, PURPOSE, AND EVALUATION (Continued)

QC Check	Definition	Purpose	Evaluation
Surrogate spike (organic analysis only) **Similar to recovery standards (Method 8280A only)**	A pure substance with properties that mimic the analyte of interest. Surrogates are compounds unlikely to be found in environmental samples and are added to samples to evaluate analytical efficiency by measuring their percent recovery. (Modified G-5 and CLP)	To assess the ability of the method to successfully recover specific non-target analytes from an actual matrix. Because surrogates are generally added to each sample in a batch, they can be used to monitor recovery on a sample-specific, rather than batch-specific basis.	Whereas the matrix spike is normally done on a batch-specific basis, the surrogate spike is done on a sample-specific basis. Taken with the information derived from other spikes (LCS, matrix spike), the bias in the analytical system can be determined.
Tuning (mass spectrometer methods only)	The analysis of a standard compound to verify that the mass spectrometer meets standard mass spectra abundance criteria prior to sample analysis. (COE)	To verify the proper working of the mass spectrometer.	Proper tuning of the mass spectrometer must be verified prior to sample analysis.

As always, project-specific requirements identified by the client supersede any requirements listed in the following tables. The requirements are meant to be the default, to be used when project-specific direction based on DQOs is not included.

Tables B-2 through B-10 are organized in most cases by instrument type. The applicable methods are specified in the table title. When there are exceptions (i.e., the QC check does not apply to all methods or instrument types in the table), they are noted in the first column of the table ("QC Check"). Each table contains the following fields (or columns):

- QC Check: The name of the QC measure that is required. If the check is only applicable to certain methods from the table, they will be noted in parentheses in this field.
- Minimum Frequency: Describes how often the QC check must be performed and, if relevant, at what point in the process (for example, prior to sample analysis). Some QC checks are only performed when another QC check fails. This will be noted in the minimum frequency field.
- Acceptance Criteria: The standard that the QC check must satisfy in order to proceed without performing corrective action. In some cases there are multiple options, all equivalently acceptable by DoD, for acceptance of a single QC check. These options will be listed and the appropriate option should be applied. There may be references to acceptance criteria published by DoD. The LCS control limits for certain methods can be found in Appendix DoD-D.
- Corrective Action: If a QC check does not meet the acceptance criteria specified in the preceding field, the corrective action field identifies what steps must be taken to ensure that the results will be valid. Requirements usually include finding the cause of failure of the acceptance criteria and rerunning the QC check. The corrective action field will also specify which other QC checks must be rerun to ensure valid data.
- Flagging Criteria: Where flagging is appropriate, the qualifier flag is listed in this field along with the criteria for using the flag. **Flagging should only be used as a last resort.** Data should only be flagged once corrective action has been performed. In many cases the field states "Flagging criteria is not appropriate." This means that corrective action must continue until the problem is solved and the QC check satisfies its acceptance criteria. Samples will not be accepted without successful completion of this QC check. This field will also specify when additional information should be detailed in the case narrative.
- Comments: This field contains further clarification of any of the previous five fields.

The following tables detail DoD-specific QC requirements for SW-846 methods, organized by instrument type:

Table B-2: Organic Analysis by Gas Chromatography and High-Performance Liquid Chromatography (methods 8011, 8015B, 8021B, 8070A, 8081A, 8082, 8141A, 8151A, 8310 and 8330
Table B-3: Organic Analysis by Gas Chromatography/Mass Spectroscopy (methods 8260B and 8270C)
Table B-4: Inorganic Analysis by Inductively Coupled Plasma (ICP) and Graphite Furnace Atomic Absorption Spectroscopy (GFAA) (methods 6010B and 7000A series)
Table B-5: Trace Metals Analysis by Inductively Coupled Plasma Mass Spectrometry (method 6020)
Table B-6: Inorganic Analysis by Colorimetric Hexavalent Chromium (method 7196A)
Table B-7: Dioxin/Furan Analysis by High-Resolution Gas Chromatography/Low-Resolution Mass Spectroscopy (method 8280A)
Table B-8: Dioxin/Furan Analysis by High-Resolution Gas Chromatography/High-Resolution Mass Spectroscopy (method 8290)
Table B-9: Cyanide Analysis (methods 9010B/9012A)
Table B-10: Common Anions Analysis (method 9056)

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015B, 8021B, 8070A, 8081A, 8082, 8141A, 8151A, 8310, AND 8330)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C)	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f)	Not applicable (NA)	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
Method detection limit (MDL) study	At initial set-up and subsequently once per 12 month period; otherwise quarterly MDL verification checks shall be performed (see box D-12)	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12)	NA	Samples cannot be analyzed without a valid MDL
Retention time window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change)	Width is ± 3 times standard deviation for each analyte retention time from 72-hour study	NA	NA	
Breakdown check (Endrin/ DDT Method 8081A only	Daily prior to analysis of samples	Degradation <15% for both Endrin and DDT	Correct problem then repeat breakdown check	Flagging criteria is not appropriate.	No samples shall be run until degradation <15%

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015B, 8021B, 8070A, 8081A, 8082, 8141A, 8151A, 8310, AND 8330) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	One of the options below (except for Method 8082, which may only use Option 1 or 3): Option 1: RSD for each analyte ≤20% Option 2: Grand mean[2] RSD ≤20%, with no individual analyte RSD >30% Option 3: linear – least squares regression: r > 0.995 Option 4: non-linear regression: coefficient of determination (COD) $r^2 \geq$ 0.990 (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration	Apply J to all analytes with RSD >20% and ≤30%. Identify in case narrative analytes with RSD >20%, provide to client the actual RSD for those analytes, and document the grand mean.	Problem must be corrected. No samples may be run until ICAL has passed. For PCB analysis, a mixture of Aroclors 1016 and 1260 is normally used to establish detector calibration linearity, unless project-specific data suggest the presence of another Aroclor (e.g., 1268, 1262). In addition, a mid level standard for each of the remaining Aroclors is analyzed for pattern recognition and response factor.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ±20% of expected value (initial source)	Correct problem and verify second source standard. If that fails then repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	At the beginning of the analytical shift	The center of the retention time window shall be set at midpoint of initial calibration curve	NA	NA	

[2] Grand mean is the average of the mean RSDs for all analytes.

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015B, 8021B, 8070A, 8081A, 8082, 8141A, 8151A, 8310, AND 8330) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window verification for each analyte and surrogate	Each calibration verification standard	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last acceptable retention time check. If they fail, redo ICAL and reset retention time window.	Flagging criteria is not appropriate for initial verification. For CCV, apply a Q-flag to all results for analytes outside the established window.	No samples shall be run without a verified retention time window at the initial verification. For method 8015B, check state methods for use of modified retention time markers with gasoline range organics (GRO) or diesel range organics (DRO).
Calibration verification (initial [ICV] and continuing [CCV])	ICV: Daily, before sample analysis. CCV: After every 10 field samples and at the end of the analysis sequence.	All analytes within ±15% of expected value (%D), or grand mean ≤15%D with no %drift/difference for any individual analyte >20%D	ICV: Correct problem, rerun ICV. If that fails, repeat initial calibration. See section 9.4.2.2.e and box 41. CCV: Correct problem then repeat CCV and reanalyze all samples since last successful calibration verification.	Identify in case narrative analytes with %D>15%, provide to client the actual %D for those analytes, and document the grand mean. ICV: Apply J to all results for analyte(s) >15% and <20% of expected range. CCV: Apply Q to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	If an individual analyte is >20% or the grand mean is >15%, no samples may be analyzed until the problem has been corrected. In Method 8021B, bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, and vinyl chloride shall be within ±20%D.
Method blank	One per preparatory batch	No analytes detected ≥ ½RL	Correct problem, then see criteria in box D-4; if required, reprep then reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch	
Laboratory control sample (LCS) containing all reported analytes	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix DoD-D)	If corrective action fails apply Q to specific analyte(s) in all samples in the associated preparatory batch	

TABLE B-2. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY (METHODS 8011, 8015B, 8021B, 8070A, 8081A, 8082, 8141A, 8151A, 8310, AND 8330) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One MS per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per every 20 project samples per matrix	RPD ≤30% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	The data shall be evaluated to determine the source of difference.
Surrogate spike (analytes identified in Appendix DoD-D)	All field and QC samples	QC acceptance criteria for LCS specified by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met. For QC samples, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (in Method 8081A, exclude toxaphene and chlordane)	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA	Apply J if RPD >40% from primary column result or Q-flag if sample is not confirmed. Discuss in the case narrative.	Report the higher of two confirmed results unless overlapping peaks are causing erroneously high results, then report the non-effected result and document in the case narrative.
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROSCOPY (METHODS 8260B AND 8270C)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C).	QC acceptance criteria published by DoD, if available; otherwise method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and subsequently once per 12 - month period; otherwise quarterly MDL verification checks shall be performed (see box D-12)	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12)	NA	Samples cannot be analyzed without a valid MDL
Tuning (MS methods only)	Prior to calibration and every 12 hours during sample analysis	Refer to method for specific ion criteria	Retune instrument and verify. Rerun affected samples.	Flagging criteria is not appropriate	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270C only)	Daily prior to analysis of samples	Degradation <20% for DDT	Correct problem then repeat breakdown check	Flagging criteria is not appropriate	No samples shall be run until degradation <20%. Benzidine and pentachlorophenol should be present at their normal responses and no peak tailing should be visible.

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROSCOPY (METHODS 8260B AND 8270C) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration for all analytes (ICAL)	Initial calibration prior to sample analysis	1. Average response factor (RF) for SPCCs: VOCs - ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs - ≥ 0.050. 2. %RSD for RFs for CCCs: VOCs and SVOCs - ≤30% and one option below; Option 1: RSD for each analyte ≤15% Option 2: Grand mean ≤15%, with no individual analyte RSD >30% Option 3: linear – least squares regression r > 0.995 Option 4: non-linear regression - coefficient of determination (COD) $r^2 \geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order)	Correct problem then repeat initial calibration	Apply J to all analytes with RSD >15% and ≤30%. Identify in case narrative analytes with RSD >15%, provide to client the actual RSD for those analytes, and document the grand mean.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration	Value of second source for all analytes within ±25% of expected value (Initial source)	Correct problem and verify second source standard. If that fails, then repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Evaluation of relative retention times (RRT)	Once per ICAL	RRT of each target analyte in each calibration standard within +/- 0.06 RRT units.	Correct problem, then rerun ICAL	Flagging criteria is not appropriate.	
Retention time window position establishment for each analyte and surrogate	Once per ICAL	Position shall be set using the midpoint standard of the initial calibration curve.	NA	NA	

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROSCOPY (METHODS 8260B AND 8270C) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Calibration verification (CV)	Daily, before sample analysis and every 12 hours of analysis time	1. Average RF for SPCCs: VOCs - ≥ 0.30 for Chlorobenzene and 1,1,2,2-tetrachlorolethane, ≥0.1 for chloromethane, bromoform, and 1,1-dichloroethane. SVOCs ≥ 0.050. 2. %Difference/Drift for CCCs: VOCs and SVOCs - ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration) In addition, DoD requires all calibration analytes within ±20%D of expected value from ICAL when using grand mean, with no individual analytes (except CCCs) >25%.	Correct problem, rerun CV. If that fails, then repeat initial calibration. See section 9.4.2.2.e and box #41.	Apply J to all results for all analytes >20%D and ≤25%D. Identify in case narrative analytes with %D > 20%, provide to client the actual %D for those analytes, and document the grand mean. Apply Q-flag if no sample material remains and analyte exceeds criteria.	
Calibration verification internal standards (CV-IS)	With every calibration verification	Retention time ±30 seconds from retention time of the midpoint standard in the ICAL EICP area within -50% to +100% of ICAL midpoint standard	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory. See corrective action for CV.	Flagging criteria is not appropriate.	Sample results are not acceptable without a valid CV-IS.
Method blank	One per preparatory batch	No analytes detected ≥ ½RL	Correct problem, then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
LCS containing all reported analytes	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient sample material is available. (see full explanation in Appendix DoD-D)	If corrective action fails, apply Q to specific analyte(s) in all samples in the associated preparatory batch	

TABLE B-3. ORGANIC ANALYSIS BY GAS CHROMATOGRAPHY/MASS SPECTROSCOPY (METHODS 8260B AND 8270C) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
MS	One MS per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One per every 20 project samples per matrix	RPD ≤30% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	The data shall be evaluated to determine the source of difference.
Surrogate spike (analytes identified in Appendix DoD-D)	All field and QC samples	QC acceptance criteria for LCS published by DoD, if available; otherwise method-specified criteria or laboratory's own in-house criteria.	For QC and field samples, correct problem, then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available.	For the specific analyte(s) in all field samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met. For QC samples, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

TABLE B-4. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) AND GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY (GFAA) (METHODS 6010B AND 7000A SERIES)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C).	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete
MDL study	At initial set-up and subsequently once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-12).	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument noise level	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12).	NA	Samples cannot be analyzed without a valid MDL
Linear range or high-level calibration check standard (ICP only)	Every 6 months	Within ±10% of expected value	NA	NA	
Initial calibration for all analytes (ICAL) **(ICP: minimum one high standard and a blank; GFAA: minimum three standards and a blank)**	Daily initial calibration prior to sample analysis	ICP: No acceptance criteria unless more than one standard is used, in which case $r \geq 0.995$. GFAA: $r \geq 0.995$	Correct problem and repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each initial calibration, prior to sample analysis	All analyte(s) within ±10% of expected value	Correct problem and verify second source standard. If that fails, then repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE B-4. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) AND GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY (GFAA) (METHODS 6010B AND 7000A SERIES) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	GFAA: within ±20% of expected value ICP: within ±10% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration.	Flagging criteria is not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Low level calibration check standard (ICP only)	Daily, after one-point initial calibration	Within ±30% of expected value	Correct problem, then reanalyze	Flagging criteria is not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Method blank	One per preparatory batch	No analytes detected ≥ ½RL	Correct problem, then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected ≥ ½RL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) in all samples associated with the blank	
Interference check solutions (ICS) (ICP only)	At the beginning of an analytical run	Within ±20% of expected value	Terminate analysis; locate and correct problem; reanalyze ICS.	Flagging criteria is not appropriate.	No samples may be analyzed without a valid ICS
LCS containing all reported analytes	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch (see full explanation in Appendix DoD-D).	If corrective action fails, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test	Each preparatory batch or when a new or unusual matrix is encountered	Five-fold dilution must agree within ±10% of the original determination	ICP: Perform post-digestion spike (PDS) addition GFAA: Perform recovery test	Flagging criteria is not appropriate.	Only applicable for samples with concentrations >50 x MDL (ICP) or >25 x MDL (GFAA)

TABLE B-4. INORGANIC ANALYSIS BY INDUCTIVELY COUPLED PLASMA (ICP) AND GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY (GFAA) (METHODS 6010B AND 7000A SERIES) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Post-digestion spike (PDS) addition (ICP only)	When dilution test fails or analyte concentration in all samples <50 x MDL	Recovery within 75-125% of expected results.	Run samples by method of standard addition (MSA) or see flagging criteria	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post-digestion spike addition	
Recovery test (GFAA only)	When dilution test fails or analyte concentration in all samples <25 x MDL	Recovery within 85-115% of expected results.	Run samples by method of standard addition (MSA) or see flagging criteria	Apply J to all sample results (for same matrix) in which MSA was not run when recovery is outside of 85-115% range	
Method of standard addition (MSA)	When matrix interference is suspected	NA	NA	NA	Document use in the case narrative.
MS	One MS per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One per every 20 project samples per matrix	RPD ≤20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	The data shall be evaluated to determine the source of difference.
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

TABLE B-5. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (METHOD 6020)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel or test method (see Appendix C).	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and once per 12 months; otherwise quarterly MDL verification checks shall be performed (see box D-12)	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument noise level	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12).	NA	Samples cannot be analyzed without a valid MDL
Instrument detection limit (IDL) study	Every 3 months	Detection limits established shall be ≤ the RLs	NA	NA	Samples cannot be analyzed without a valid IDL
Tuning (MS methods only)	Prior to initial calibration	Per 6020 (5.8)	Retune instrument then reanalyze tuning solutions	Flagging criteria is not appropriate.	No analysis shall be performed without a valid MS tune.
Initial calibration (ICAL) **(minimum one high standard and a blank)**	Daily initial calibration prior to sample analysis	If more than one calibration standard is used, r≥ 0.995	Correct problem, then repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analytes within ±10% of expected value (initial source)	Correct problem and verify second source standard. If that fails, then repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 samples and at the end of the analysis sequence	All analytes within ±10% of expected value	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration. Reanalyze all samples since the last successful calibration.	Flagging criteria is not appropriate.	Problem must be corrected. Results may not be reported without a valid CCV.
Low-level calibration check standard	Daily, after one-point initial calibration	Within ±30% of expected value	Correct problem, then reanalyze	Flagging criteria is not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.

TABLE B-5. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (METHOD 6020) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Linear range or high-level calibration check standard	Every 6 months	Within ±10% of expected value	NA	NA	
Method blank	One per preparatory batch	No analytes detected ≥½ RL	Correct problem, then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch	
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence	No analytes detected ≥½ RL	Correct problem, then reprep and reanalyze calibration blank and previous 10 samples	Apply B to all results for specific analyte(s) in all samples associated with the blank	
Interference check solutions (ICS-A and ICS-AB)	At the beginning and end of an analytical run or twice during a 12-hour period, whichever is more frequent	ICS-A: All non-spiked analytes < RL (unless they are a verified trace impurity from one of the spiked analytes) ICS-AB: Within ±20% of true value	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all affected samples	If corrective action fails, apply Q to all results for specific analyte(s) in all samples associated with the ICS	
LCS containing all reported analytes	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix DoD-D)	If corrective action fails, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	
Dilution test	Each preparatory batch	Five-fold dilution must agree within ±10% of the original measurement	Perform post-digestion spike addition	Flagging criteria is not appropriate.	Only applicable for samples with concentrations >100 x MDL
Post digestion spike addition	When dilution test fails or analyte concentration for all samples <100 x MDL	Recovery within 75-125% of expected results	Run samples by method of standard addition (MSA) or see flagging criteria	Apply J to all sample results (for same matrix) for specific analyte(s) for all samples associated with the post - digestion spike addition	
Method of standard additions (MSA)	When matrix interference is suspected	NA	NA	NA	Document use in the case narrative

TABLE B-5. TRACE METALS ANALYSIS BY INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (METHOD 6020) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
MS	One MS per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD or sample duplicate	One per every 20 project samples per matrix	RPD < 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	The data shall be evaluated to determine the source of difference.
Internal standards (IS)	Every sample	IS intensity within 80-120% of intensity of the IS in the initial calibration	Perform corrective action as described in method 6020 (8.3)	Flagging criteria is not appropriate	
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

TABLE B-6. INORGANIC ANALYSIS BY COLORIMETRIC HEXAVALENT CHROMIUM (METHOD 7196A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel or test method (see Appendix C).	QC acceptance criteria published in method; otherwise QC acceptance criteria established in-house by laboratory.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete
MDL study	At initial set-up and subsequently once per 12-month period	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument noise level	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12).	NA	Samples cannot be analyzed without a valid MDL
Reference blank (reagent water)	Before beginning standards or sample analysis	NA	NA	NA	Used for blank subtraction of standards, field and QC samples. For turbid field samples, a turbidity blank must be used instead of the reference blank (using a sample aliquot prepped in accordance with 7196A (7.1))
Initial calibration (ICAL) **(minimum three standards and a blank)**	Daily initial calibration prior to sample analysis	r$0.995	Correct problem and repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed
Second source calibration verification **(also known as independently prepared check standard)**	Before beginning a sample run and after every 15 samples.	Value of second source for all analytes within ±10% of expected value (initial source)	Correct problem and verify second source standard. If that fails, then repeat calibration and reanalyze all samples since last successful calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE B-6. INORGANIC ANALYSIS BY COLORIMETRIC HEXAVALENT CHROMIUM (METHOD 7196A) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample matrix verification **(also known as matrix spike)**	Once for every sample matrix analyzed	Spike recovery within 85-115%	If check indicates interference, dilute and reanalyze sample; persistent interference indicates the need to use alternative method or analytical conditions, or to use method of standard additions.	Flagging criteria is not appropriate.	Verification check ensures lack of reducing condition or interference from matrix. Additional corrective actions are identified in method 7196A (7.4 and 7.5).
Method blank	One per preparatory batch	No analytes detected ≥ ½RL	Correct problem then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch	
LCS	One LCS per preparatory batch	QC acceptance criteria specified by DoD; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix DoD-D).	If corrective action fails, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	
MSD or sample duplicate	One per every 10 project samples per matrix	RPD ≤ 30% (between MS and MSD or sample and sample duplicate)	Examine project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria not met	Refer to sample matrix verification sample for MS data evaluation
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

TABLE B-7. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROSCOPY (METHOD 8280A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C).	QC acceptance criteria established in-house by laboratory.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL study	At initial set-up and quarterly MDL verification checks shall be performed (see box D-12).	See 40 CFR 136B. MDL verification check must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12)	NA	Samples cannot be analyzed without a valid MDL. Refer to Sample Estimated Detection Limit.
Tuning (MS methods only)	Prior to calibration standards	Verify MS calibration per 8280A (7.13.1).	Retune instrument; verify.	Flagging criteria is not appropriate.	
Retention time window defining mix	At method set-up and prior to calibration standards	Verify descriptor switching times per 8280A (7.13.2).	Correct problem then repeat retention time window defining mix	Flagging criteria is not appropriate.	
GC column performance check	Prior to initial calibration or calibration verification standards for each 12-hour period of sample analysis.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of #25%, per 8280A (7.12.2)	Correct problem, then repeat column performance check	Flagging criteria is not appropriate.	Needed only if using a column other than DB-5.
Column performance check for DB-5 columns	Included with the ICAL standard (CC3) and the calibration verification standard analyses	<u>Peak separation of standard CC3:</u> Peak between the 2,3,7,8-TCDD and 1,2,3,4-TCDD must be resolved with a valley of #25%, per 8280A (7.12.1). <u>For calibration verification standard only:</u> Peak separation between 1,2,3,4,7,8-HxCDD and 1,2,3,6,7,8-HxCDD must be resolved with a valley of #50%, per 8280A (7.13.3.6.1)	Correct problem, then repeat column performance check	Flagging criteria is not appropriate.	

TABLE B-7. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROSCOPY (METHOD 8280A) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration for all analytes identified in Table 1 of Method 8280A (ICAL)	Initial calibration prior to sample analysis and as needed by the failure of calibration verification standard.	Ion abundance ratios in accordance with 8280A Table 9 (7.13.3.1.1) and %RSD ≤15% for labeled IS and unlabeled PCDD/PCDF RRFs per 8280A (7.13.3.4)	Correct problem, then repeat initial calibration	Apply Q to all analytes with RSD >15%.	Problem must be corrected. No samples may be run until ICAL has passed.
Calibration verification **(Table 4 of Method 8280A – final concentrations of Standard CC3 of Table 1)**	At the beginning of each 12-hour period of sample analysis, after successful GC and MS resolution checks.	Ion abundance in Table 9 of 8280A must be met for all PCDD/PCDF peaks, including labeled IS and recovery standards, and Sensitivity criteria of an S/N ratio >2.5 for unlabeled PCDD/PCDF ions and >10 for labeled IS and recovery standards per 8280A (7.13.3.6.3) and RF within 30% (% difference) of mean RF from initial calibration	Correct problem, rerun calibration verification. If that fails, then repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration verification has passed.
Sensitivity check **(Standard CC1 of Table 1 of Method 8280A)**	End of 12-hour sample analysis period (Injection must be done within the 12-hour period.)	See criteria for retention time check, ion abundances, and S/N ratios noted above for calibration verification standard per 8280A (7.13.3.7)	Correct problem, then repeat calibration and reanalyze samples indicating a presence of PCDD/PCDF less than quantitation limit or when maximum possible concentration is reported	Flagging criteria is not appropriate.	Nondetects and samples with positive results above the method quantitation limit do not need to be reanalyzed.
Method blank	One per preparatory batch	No analytes detected ≥ MDL for the analyte or ≥5% of the associated regulatory limit for the analyte or ≥5% of the sample result for the analyte, whichever is greater, per 8280A (8.4.3)	Correct problem, then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to the result for specific analyte(s) in all samples in the associated preparatory batch	

TABLE B-7. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROSCOPY (METHOD 8280A) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing analytes identified in Table 5 of Method 8280A	One LCS per preparatory batch	QC acceptance criteria specified by DoD; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for the failed analytes in all samples in the associated preparatory batch (see full explanation in Appendix DoD-D)	If corrective action fails or if insufficient sample is available for reanalysis, apply Q to specific analytes in all samples in the associated preparatory batch.	LCS compounds are the same as the MS compounds identified in Table 5 of Method 8280A (8.4.2)
MS containing analytes identified in Table 5 of Method 8280A	One MS per every 20 project samples per matrix (see box D-6)	For evaluation of MS, use QC acceptance criteria specified by DoD for LCS	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	Check other QC measures to verify matrix interference. For instance, verify that the LCS shows control of the batch analysis. Also verify sample recoveries for the internal standards, recovery, and cleanup standards for an indication of potential impact.
MSD or sample duplicate	One per every 20 project samples per matrix	RPD ≤ 20% (between MS and MSD or sample and sample duplicate)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if criteria are not met.	MSD spike includes the MS compounds identified in Table 5 of Method 8280A.
Internal standards identified in Table 3 of Method 8280A	Every sample, standard, and QC sample	% recovery for each IS in the original sample (prior to any dilutions) must be within 25-150% per 8280A (7.15.5.2)	Correct problem, then reprep and reanalyze the sample(s) with failed IS	Apply Q to results of all affected samples	

TABLE B-7. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROSCOPY (METHOD 8280A) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample PCDD/PCDF identification	Verify all positive sample detections per 8280A (7.14.5)	Verify that absolute RT at maximum height is within -1 to +3 secs. of corresponding labeled standard, or the RRT of analytes is within 0.05 RT units of the calibration verification standard, or is within the RT window established with the RT window defining mix for the corresponding homologue per 8280A (7.14.5.1) and Absolute RTs of the two recovery standards must be "10 sec. of the calibration verification standard (7.14.5.1) and All ions listed in Table 8 of Method 8280A must be present in the SICP (7.14.5.2) and S/N ratio of ISs $10 times background noise and must have not saturated the detector. Remaining ions on Table 8 must have an S/N ratio $2.5 times the background noise (7.14.5.3) and Ion abundance in Table 9 of 8280A must be met for all ISs, recovery, and cleanup standards (7.14.5.4) and No signal is present having an S/N ratio > 2.5 times background for the corresponding ether (PCDPE) detected at the same RT (" 2 sec.) (7.14.5.5)	Correct problem, then reprep and reanalyze the sample(s) with failed criteria for any of the internal, recovery, or cleanup standards. If PCDPE is detected or if sample peaks present do not meet all identification criteria, calculate the EMPC (estimated maximum possible concentration) according to 8280A (7.15.7).	Flagging criteria is not appropriate.	

TABLE B-7. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/LOW-RESOLUTION MASS SPECTROSCOPY (METHOD 8280A) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample estimated detection limit (EDL)	Every sample that indicates nondetects or detections that are less than 2.5 times background noise	Per 8280A (7.15.6)	NA	NA	
Sample estimated maximum possible concentration (EMPC)	Every sample that indicates a detection ≥ 2.5 times S/N ratio.	Identification criteria in 7.4.5 of 8280A must be met, and response for both quantitation ions must be ≥ 2.5 times S/N ratio of background (7.15.7)	NA	NA	
Sample 2,3,7,8-TCDD toxicity equivalents (TE) concentration	All positive detections	Per 8280A (7.15.8)	NA	NA	Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD.
Results reported between MDL and RL	Positive detections calculated per 8280A (7.15.1)	NA	NA	Apply J to all results between MDL and RL	

TABLE B-8. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROSCOPY (METHOD 8290)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C).	QC acceptance criteria established in-house by laboratory.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete
MDL study	At initial set-up and quarterly MDL verification checks shall be performed (see box D-12).	See 40 CFR 136B. MDL verification check must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12)	NA	Samples cannot be analyzed without a valid MDL. Refer to Sample EDL.
Tuning (MS methods only)	At the beginning and the end of each 12-hour period of analysis	Static resolving power ≥ 10,000 (10% valley) for identified masses per 8290 (7.6.2.2 and 8.2.2.1/8.2.2.3), and Monitor mass drift of lock-mass ion per 8290 (8.2.2.2)	Retune instrument; verify. Rerun affected samples.	Flagging criteria is not appropriate.	Recommend that a check of static resolution also be documented before and after each analysis.
GC column performance check	Prior to initial calibration or calibration verification standards for each 12-hour period of sample analysis.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of #25% per 8290 (8.2.1.2) and Identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram (8.2.1.2) and Absolute retention times for switching times for all components <10 sec. (8.2.1.3)	Correct problem then repeat column performance check	Flagging criteria is not appropriate.	

TABLE B-8. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROSCOPY (METHOD 8290) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration for all analytes identified in Table 5 of Method 8290 (ICAL)	Initial calibration prior to sample analysis, as needed by the failure of calibration verification standard, and when a new lot is used for standard source of CCV, sample fortification solution (IS), and recovery standards.	Ion abundance ratios in accordance with criteria in Table 8 of Method 8290 (7.7.1.4.1/7.7.2.3) and S/N ratio ≥ 10 for all ions (7.7.2.2) and %RSD ≤20% for 17 unlabeled standards and %RSD ≤ 30% for the 9 labeled IS mean RFs (7.7.2.1)	Correct problem, then repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Routine (continuing) calibration check **(Table 5 of 8290 - final concentrations of HRCC3)**	At the beginning of each 12-hour period after successful GC resolution and mass resolution checks, and at the end of 12-hour shift.	Ion abundance ratios in accordance with criteria in Table 8 of Method 8290 (7.7.4.3) and RF within 20% D for unlabeled standards from mean RF from initial calibration (7.7.4.1) and RF within 30% D for labeled standards from mean RF from initial calibration (7.7.4.2) End-of-run CCV only: RF within 25% D for unlabeled standards from mean RF from initial calibration and RF within 35% D for labeled standards from mean RF from initial calibration (8.4.2.4) requires the use of the mean RF from the two daily CCVs instead of the ICAL mean RF value.	Correct problem, repeat calibration verification standard one more time. If that fails, then repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification. Evaluation of corresponding labeled/unlabeled standards may impact the corrective action required (see 8290 section 7.7.4.4) If ending CCV RF > 25% and 35% for unlabeled and labeled standards, respectively, a new ICAL must be run immediately (within 2 hr.). Reanalyze samples with positive detections, if necessary.	Flagging criteria is not appropriate for routine calibration check. Q-flag (noncompliances) for end-of-run continuing calibration check.	Problem must be corrected. No samples may be run until routine (beginning of 12-hour period) calibration verification has passed.

TABLE B-8. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROSCOPY (METHOD 8290) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch, run after calibration standards and before samples	No analytes detected ≥ ½RL	Correct problem then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to the result for specific analyte(s) in all samples in the associated preparatory batch	
Field blanks and/or rinsates	Per project requirements (see 8290 section 8.3.4)	Per project requirements	Per project requirements	Per project requirements	
Performance evaluation (PE) sample	Per project requirements (see 8290 section 8.3.1)	Per project requirements	Correct problem, then reprep and reanalyze the PE and all samples in the associated batch for failed analytes in all samples in the associated batch, if sufficient sample material is available.	If corrective action fails, apply Q to specific analyte(s) in all samples in the associated preparatory batch.	
MS	One MS per every 20 project samples per matrix (see box D-6)	QC acceptance criteria for lab's in-house control limits.	Check other QC measures to verify matrix interference. For instance, verify that the PE sample shows control of the batch analysis. Also verify sample recoveries for the internal, recovery and cleanup standards for an indication of potential impact.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	MS spike includes all compounds identified in Table 5 of Method 8290 at the concentration corresponding to HRCC3 standard.
MSD or sample duplicate	One per every 20 project samples per matrix	RPD ≤ 25% for laboratory duplicates per 8290 (8.3.5.1.1); RPD ≤ 20% for MS/MSD (8.3.6.4)	Refer to MS.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met	MSD spike includes all compounds identified in Table 5 of Method 8290 at the concentration corresponding to HRCC3 standard.
Internal standards identified in Table 2 of Method 8290	Every sample, standard, and QC sample	%Recovery for each IS in the original sample (prior to dilutions) must be within 40-135% per 8290 (8.4)	Correct problem, then reprep and reanalyze the sample(s) with failed IS.	Apply Q to results of all affected samples.	
Sample PCDD/PCDF identification	Verify all sample positive detections per 8290 (7.8.4)	Retention time of sample components in accordance with stated criteria in 8290 (7.8.4.1)	Correct problem, then reprep and reanalyze the sample(s) with failed criteria for any of the internal, recovery, or	Flagging criteria is not appropriate.	Positive identification of 2,3,7,8-TCDF on the DB-5 or equivalent column must be reanalyzed on a column

TABLE B-8. DIOXIN/FURAN ANALYSIS BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/HIGH-RESOLUTION MASS SPECTROSCOPY (METHOD 8290) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample PCDD/PCDF identification (continued)		and Ion abundance ratios in accordance with criteria on Table 8 of 8290 (7.8.4.2), and S/N Ratio of all ions $2.5 times background noise (7.8.4.3) and No signal present having a S/N ratio > 2.5 for the corresponding ether (PCDPE) detected at the same retention time ("2 sec) (7.8.4.4)	cleanup standards. If PCDPE is detected or if sample peaks present do not meet all identification criteria, calculate the EMPC (estimated maximum possible concentration) according to 8290 (7.9.5.2.1)		capable of isomer specificity (DB-225) (see 8290 section 3.4)
Sample estimated detection limit (EDL)	Every sample that indicates nondetects or detections that are < 2.5 times background noise	Per 8290 (7.9.5)	NA	NA	
Sample estimated maximum possible concentration (EMPC)	Every sample that indicates a detection ≥ 2.5 times S/N response	Identification criteria in 8290 (7.4.5) must be met, and response for both quantitation ions must be ≥ 2.5 times S/N ratio for background (7.9.5.2.1)	NA	NA	
Sample 2,3,7,8-TCDD toxicity equivalents (TE) concentration	All positive detections	Per 8290 (7.9.7)	NA	NA	Recommended reporting convention by the EPA and CDC for positive detections in terms of toxicity of 2,3,7,8-TCDD
Results reported between MDL and RL	Positive detections calculated per 8290 (7.9.1)	NA	NA	Apply J to all results between MDL and RL	

TABLE B-9. CYANIDE ANALYSIS (METHODS 9010B/9012A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C).	QC acceptance criteria published by DoD, if available; otherwise use method-specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is complete
MDL study	At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-12).	See 40 CFR 136B. MDL verification check must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12)	NA	Samples cannot be analyzed without a valid MDL
Multipoint calibration curve **(six standards and a calibration blank)**	Initial daily calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem, then repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has passed.
Distilled standards **(one high and one low)**	Once per multipoint calibration	Within ±10% of true value	Correct problem, then repeat distilled standards	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until distilled standards have passed.
Second source calibration verification check standard	Once per stock standard preparation	Value of second source within ±15% of expected value (initial source).	Correct problem and verify second source standard. If that fails, then repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Method blank	One per preparatory batch	No analytes detected ≥ ½RL	Correct problem, then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch.	
LCS containing all reported analytes	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated preparatory batch, if sufficient sample material is available (see full explanation in Appendix DoD-D).	If corrective action fails apply Q to the specific analyte in all samples in the associated preparatory batch.	

TABLE B-9. CYANIDE ANALYSIS (METHODS 9010B/9012A) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
MS	One per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD	One per every 20 project samples per matrix	RPD ≤20% (between MS or MSD)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample duplicate	Once per every 20 project samples	%D of duplicate within ± 20% of sample	Correct problem and reanalyze sample and duplicate	Apply Q if sample cannot be rerun or reanalysis does not correct problem.	
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

TABLE B-10. COMMON ANIONS ANALYSIS (METHOD 9056)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, or test method (see Appendix C).	QC acceptance criteria published by DoD, if available; otherwise use method-specified criteria	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see section C.1.f).	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check sample (e.g., LCS or PT sample). No analysis shall be allowed by analyst until successful demonstration of capability is completed.
MDL study	At initial set-up and subsequently once per 12-month period; otherwise quarterly MDL verification checks shall be performed (see box D-12).	See 40 CFR 136B. MDL verification checks must produce a response at least 3 times greater than instrument's noise level.	Run MDL verification check at higher level and higher MDL set or reconduct MDL study (see box D-12).	NA	Samples cannot be analyzed without a valid MDL
Retention time window width calculated for each analyte	After method set-up and after major maintenance (e.g., column change)	Width is ± 3 times standard deviation for each analyte retention time over 8-hour period	NA	NA	
Multipoint calibration for all analytes **(minimum three standards and one calibration blank)**	Initial calibration prior to sample analysis	Correlation coefficient ≥0.995 for linear regression	Correct problem, then repeat initial calibration	Flagging criteria is not appropriate.	Problem must be corrected. No sample may be run until calibration has passed.
Second source calibration verification	Once after each multipoint calibration	Value of second source for all analytes within ±10% of expected value (initial source).	Correct problem and verify second source standard. If that fails, then repeat initial calibration.	Flagging criteria is not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Retention time window position establishment for each analyte	Once per multipoint calibration	Position shall be at midpoint of calibration curve	NA	NA	
Retention time window verification for each analyte	Each calibration verification	Analyte within established window	Correct problem, then reanalyze all samples analyzed since the last retention time check. If they fail, redo ICAL and reset retention time window.	Flagging criteria is not appropriate.	No samples shall be run without a verified retention time window.

TABLE B-10. COMMON ANIONS ANALYSIS (METHOD 9056) (Continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial calibration verification (ICV)	Daily before sample analysis; and when eluent is changed, and with every batch of samples	All analytes within ±10% of expected value and retention times within appropriate windows	Correct problem, rerun ICV. If that fails, then repeat initial calibration (see section 9.4.2.2.e and box #41).	Flagging criteria is not appropriate.	No samples may be run without verifying initial calibration
Midrange continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence	Instrument response within ±5% of expected response	Correct problem, then repeat continuing calibration verification and reanalyze all samples since last successful calibration verification	Apply Q to all results for the specific analyte(s) in all samples since the last acceptable calibration verification	
Method blank	One per preparatory batch	No analytes detected ≥ ½RL	Correct problem, then see criteria in box D-4. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	Apply B to all results for the specific analyte(s) in all samples in the associated preparatory batch	
LCS containing all reported analytes	One LCS per preparatory batch	QC acceptance criteria specified by DoD, if available; see box D-5 and Appendix DoD-D.	Correct problem then reprep and reanalyze the LCS and all samples in the associated batch for failed analytes in all samples in the associated prepatory batch, if sufficient sample material is available.	If corrective action fails apply Q to specific analyte(s) in all samples in the associated preparatory batch	
MS	One MS per every 20 project samples per matrix (see box D-6)	For matrix evaluation, use QC acceptance criteria specified by DoD for LCS.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
MSD	One per every 20 project samples per matrix	RPD ≤20% (between MS and MSD)	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in all samples collected from the same site matrix as the parent, apply J if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Sample duplicate	One per every 10 samples	%D ≤10%	Correct problem and reanalyze sample and duplicate	If corrective action fails, apply Q to specific analyte(s) in the sample	
Results reported between MDL and RL	NA	NA	NA	Apply J to all results between MDL and RL	

ACRONYMS FOR APPENDIX DOD-B

CC3: The third of five solutions for instrument calibration used in method 8280A
CCC: Calibration check compounds
CCV: Continuing calibration verification
CFR: Code of Federal Regulations
COD: Coefficient of determination
COE: Army Corps of Engineers
CV: Calibration verification
CV-IS: Calibration verification of internal standards
D: Difference or drift
DDT: 2,2-bis(p-chlorophenyl)-1,1,1-trichloroethane/dichlorodiphenyl-trichloroethane/p,p'-DDT
DoD: Department of Defense
DQO: Data quality objective
DRO: Diesel range organics
EDL: Estimated detection limit
EICP: Extraction ion current profile
GC: Gas chromatography
GC/MS: Gas chromatography with subsequent mass spectrometry
GFAA: Graphite furnace atomic absorption spectrometry
GRO: Gasoline range organics
HPLC: High performance liquid chromatography
HxCDD: Hexachlorodibenzo-p-dioxin (solution used for calibration verification)
ICAL: Initial calibration
ICP: Inductively coupled plasma
ICP/MS: Inductively coupled plasma with subsequent mass spectrometry
ICS: Interference check solution
ICV: Initial Calibration Verification
IS: Internal standard
IDL: Instrument detection limit
LCS: Laboratory control sample
MDL: Method detection limit
MS: Mass spectrometry
MS: Matrix spike
MSA: Method of standard additions
MSD: Matrix spike duplicate
PCB: Polychlorinated biphenyl
PCDD: Polychlorinated dibenzodioxin
PCDF: Polychlorinated dibenzofuran
PDS: Post-digestion spike
PE: Performance evaluation
PT: Proficiency testing
QC: Quality control
QSM: Quality Systems Manual
RF: Response factor
RL: Reporting limit
RPD: Relative percent difference
RRT: Relative retention time
RSD: Relative standard deviation
RT: Retention time
SICP: Selected ion current profile
S/N: Signal to noise ratio
SPCC: System performance check compound
SVOC: Semi-volatile organic compound

TCDD: Tetrachlorodibenzo-p-dioxin
TCDF: Tetrachlorodibenzofuran
VOC: Volatile Organic Compound

GLOSSARY FOR APPENDIX DOD-B

Aliquot: A discrete, measured, representative portion of a sample taken for analysis. (Team; EPA QAD glossary)

Analyte: The specific chemicals or components for which a sample is analyzed; may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA glossary)

Atomization: A process in which a sample is converted to free atoms. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry.1992)

Congener: A member of a class of related chemical compounds (e.g., PCBs, PCDDs).

Digestion: A process in which a sample is treated (usually in conjunction with heat) to convert the sample to a more easily measured form.

Duplicate: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Eluent: A solvent used to carry the components of a mixture though a stationary phase. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

Elute: To extract; specifically, to remove (adsorbed material) from an adsorbent by means of a solvent. (Merriam-Webster's Collegiate Dictionary, 2000)

Elution: A process in which solutes are washed though a stationary phase by the movement of a mobile phase. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

False Negative: An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.

False Positive: An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

Homologue: One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, CH_3OH (methanol), C_2H_5OH (ethanol), C_3H_7OH (propanol), C_4H_9OH (butanol), etc., form a homologous series. (The Condensed Chemical Dictionary G.G.Hawley, ed. 1981)

Interference, spectral: Occurs when particulate matter from the atomization scatters the incident radiation from the source or when the absorption or emission of an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

Interference, chemical: Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

Internal Standard: A pure substance that is introduced in known amount into each calibration standard and field and QC sample of the analyte. The ratio of the analyte signal to the internal standard signal is then used to determine the analyte concentration. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

Isomer: Generally, any two chemicals with the same chemical formula but a different structure. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane. (http://www.kcpc.usyd.edu.au/discovery/glossary-all.html)

Matrix: The collection of all of the various constituents making up an analytical sample. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Method of Standard Additions: A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Retention Time: The time between sample injection and the appearance of a solute peak at the detector. (Skoog, West, and Holler. Fundamentals of Analytical Chemistry. 1992)

Signal to Noise Ratio: The signal carries information about the analyte, while noise is made up of extraneous information that is unwanted because it degrades the accuracy and precision of an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

Standard: Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by the US National Institute of Standards and Technology (NIST) and characterized for absolute content, independent of analytical test method.

APPENDIX DOD-C – TARGET ANALYTE LISTS

The lists of analytes provided in this appendix are to be used as a default whenever no analyte list has been provided by the client. It is anticipated that project-specific information will identify the analyses needed. The client may also identify specific analytes associated with those analyses. If not, the following target analyte lists shall be the default for those analyses identified as appropriate to the site. This appendix is not needed when DoD personnel have used site-specific information to identify project-specific target analytes. If limited site-specific information is available, the target analyte lists may be used as a baseline list from which the client may add or subtract specific analytes. The following target analyte lists were compiled by all three DoD components to include common analytes of concern at DoD sites as well as the Superfund list of 110 most frequently occurring chemicals.

The analytes are organized by SW-846 methods. The organization is for convenience only and is not meant to infer that the laboratory must conduct the analysis with these specific methods. The project-specific QAPP will identify the analytical method to be used, and the target analytes may be carried over to those different methods. In several cases one analyte may be detected by multiple methods. The comment field in each table identifies what alternative SW-846 method(s) can detect the analyte. The following tables list the default DoD target analytes for SW-846 methods commonly used by DoD:

SW-846 Methods

Although the target analyte lists in this appendix are organized by SW-846 methods, the laboratory is not restricted to those methods when conducting analyses. In addition, this appendix refers to the method versions current at the time of publication. As methods are updated subsequent versions of this manual will incorporate the changes. If the tables in this appendix do not yet correspond with the most recent version of the SW-846 method, or if a new method that analyzes for the same group of analytes becomes available, the target analyte lists in this appendix still apply.

Table C-1: Method 8260B (volatile organic compounds)
Table C-2: Method 8270C (semivolatile organic compounds)
Table C-3: Methods 8280A and 8290 (dioxins/furans)
Table C-4: Method 8141A (organophosphorus pesticides)
Table C-5: Method 8151A (phenoxyacid herbicides)
Table C-6: Method 8310 (polynuclear aromatic hydrocarbons)
Table C-7: Method 8330 (explosives)
Table C-8: Method 8081A (organochlorine pesticides)
Table C-9: Method 8082 (polychlorinated biphenyls)
Table C-10: Methods 6010B, 7000A series, 9010B, 9012A, and 9056 (inorganics).

[Note: Analytes often have many synonyms; refer to the CAS number when there is uncertainty regarding an analyte name.]

During a multi-laboratory study of laboratory control sample (LCS) recoveries, several compounds on the target analyte lists were identified as poor performing analytes for certain methods. These analytes are included in the target analyte lists in the following tables since they should be included in the calibration standard; however, they should be treated separately in the LCS. For further explanation on how to treat poor performing analytes when they are detected in the calibration or are target analytes of concern see Appendix DoD-D. The analytes are identified as poor performers on the following tables. For methods 8151A and 8330, results will be published following further analysis. No data were gathered for the following methods; therefore, no conclusions on analyte performance can be made: 8280A, 8290, 8141A, 7000A series (GFAA), 9010B, 9012A and 9056.

TABLE C-1. SW-846 METHOD 8260B (VOLATILE ORGANIC COMPOUNDS) TARGET ANALYTE LIST[3]

Volatile Organic Compound	CAS #	Comments	Volatile Organic Compound	CAS #	Comments
Acetone	67-64-1		Trans-1,3-Dichloropropene	10061-02-6	
Benzene	71-43-2		Ethylbenzene	100-41-4	
Bromobenzene	108-86-1		2-Hexanone	591-78-6	
Bromochloromethane	74-97-5		Hexachlorobutadiene	87-68-3	
Bromodichloromethane	75-27-4		Isopropylbenzene	98-82-8	
Bromoform	75-25-2		p-Isopropyltoluene	99-87-6	
Bromomethane (Methyl bromide)	74-83-9		Methylene chloride	75-09-2	
2-Butanone (MEK)	78-93-3		4-Methyl-2-pentanone (MIBK)	108-10-1	
n-Butylbenzene	104-51-8		Methyl Tert-butyl Ether (MTBE)	1634-04-4	
sec-Butylbenzene	135-98-9		Naphthalene	91-20-3	See also 8270C and 8310
tert-Butylbenzene	98-06-6		n-Propylbenzene	106-65-1	
Carbon disulfide	75-15-0		Styrene	100-42-5	
Carbon tetrachloride	56-23-5		1,1,1,2-Tetrachloroethane	630-20-6	
Chlorobenzene	108-90-7		1,1,2,2-Tetrachloroethane	79-34-5	
Chlorodibromomethane[4]	124-48-1		Tetrachloroethene	127-18-4	
Chloroethane	75-00-3		Toluene	108-88-3	
Chloroform	67-66-3		1,2,3-Trichlorobenzene	87-61-6	
Chloromethane	74-87-3		1,2,4-Trichlorobenzene	120-82-1	
2-Chlorotoluene	95-49-8		1,1,1-Trichloroethane	71-55-6	
4-Chlorotoluene	106-43-4		1,1,2-Trichloroethane	79-00-5	
1,2-Dibromo-3-chloropropane	96-12-8		Trichloroethene	79-01-6	
1,2-Dibromoethane (Ethylene dibromide)	106-93-4		Trichlorofluoromethane	75-69-4	
Dibromomethane	74-95-3		1,2,3-Trichloropropane	96-18-4	
1,2-Dichlorobenzene	95-50-1	See also 8270C	1,2,4-Trimethylbenzene	95-63-6	
1,3-Dichlorobenzene	541-73-1	See also 8270C	1,3,5-Trimethylbenzene	108-67-8	
1,4-Dichlorobenzene	106-46-7	See also 8270C	Vinyl chloride	75-01-4	
Dichlorodifluoromethane	75-71-8		o-Xylene	95-47-6	
1,1-Dichloroethane	75-34-3		m,p-Xylene	108-38-3/ 106-42-3	
1,2-Dichloroethane	107-06-2		Xylene (total)[5]	1330-20-7	
1,1-Dichloroethene	75-35-4		4-Bromofluorobenzene	460-00-4	Surrogate
Cis-1,2-Dichloroethene	156-59-2		Dibromofluoromethane	1868-53-7	Surrogate

[3] Vinyl acetate has often been included on DoD target analyte lists for method 8260B in the past. Data indicate that it may not consistently produce quantitative data with this method. Therefore, it has purposely been removed from the target analyte list. The compound may be added back to the list on a project-specific basis.
[4] Though not selected by DoD, this compound was retained due to its inclusion on the Superfund list of 110 most frequently occurring chemicals.
[5] Data may be reported on a project-specific basis as Total Xylene, however, for purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene.

TABLE C-1. SW-846 METHOD 8260B (VOLATILE ORGANIC COMPOUNDS) TARGET ANALYTE LIST (Continued)

Volatile Compound	CAS #	Comments	Volatile Compound	CAS #	Comments
trans-1,2-Dichloroethene	156-60-5		1,2-Dichlorobenzene-d4	2199-69-1	Surrogate
1,2-Dichloropropane	78-87-5		1,2-Dichloroethane-d4	17060-07-0	Surrogate
1,3-Dichloropropane	142-28-9		Fluorobenzene	462-06-6	Surrogate
2,2-Dichloropropane	594-20-7		Toluene-d8	2037-26-5	Surrogate
1,1-Dichloropropene	563-58-6		Pentafluorobenzene	363-72-4	Surrogate
cis-1,3-Dichloropropene	10061-01-5				

TABLE C-2. SW-846 METHOD 8270C (SEMIVOLATILE ORGANIC COMPOUNDS) TARGET ANALYTE LIST[6]

Semivolatile Compound	CAS #	Comments	Semivolatile Compound	CAS #	Comments
Acenaphthene	83-32-9	See also 8310	2,4-Dinitrotoluene	121-14-2	
Acenaphthylene	208-96-8	See also 8310	2,6-Dinitrotoluene	606-20-2	
Anthracene	120-12-7	See also 8310	1,2-Diphenylhydrazine	122-66-7	
Benzidine	92-87-5		Di-n-octyl phthalate	117-84-0	
Benzoic acid[7,8]	65-85-0		Fluoranthene	206-44-0	See also 8310
Benz(a)anthracene	56-55-3	See also 8310	Fluorene	86-73-7	See also 8310
Benzo(b)fluoranthene	205-99-2	See also 8310	Hexachlorobenzene	118-74-1	
Benzo(k)fluoranthene	207-08-9	See also 8310	Hexachlorobutadiene	87-68-3	
Benzo(g,h,i)perylene	191-24-2	See also 8310	Hexachloroethane	67-72-1	
Benzo(a)pyrene	50-32-8	See also 8310	Indeno(1,2,3-cd)pyrene	193-39-5	See also 8310
Benzyl alcohol	100-51-6		Isophorone	78-59-1	
Bis(2-chlorethoxy)methane	111-91-1		2-Methylnaphthalene	91-57-6	
Bis(2-chloroethyl) ether	111-44-4		2-Methylphenol	95-48-7	
Bis(2-chloroisopropyl) ether	108-60-1		3-Methylphenol/4-Methylphenol	108-39-4 / 106-44-5	
Bis(2-ethylhexyl) phthalate	117-81-7		Naphthalene	91-20-3	See also 8260B, 8310
4-Bromophenyl phenyl ether	101-55-3		2-Nitroaniline	88-74-4	
Butyl benzyl phthalate	85-68-7		3-Nitroaniline	99-09-2	
Carbazole	86-74-8		4-Nitroaniline	100-01-6	
4-Chloroaniline[7]	106-47-8		Nitrobenzene	98-95-3	See also 8330
4-Chloro-3-methylphenol	59-50-7		2-Nitrophenol	88-75-5	

[6] Hexachlorocyclopentadiene has often been included on DoD target analyte lists for method 8270C in the past. The analyte is an intermediate product of pesticide manufacturing and would not be expected to be found on a DoD site. Therefore, it has purposely been removed from the target analyte list. If pesticide manufacturing has occurred at the site, the compound should be added back in on a project-specific basis.

[7] Poor performing analyte for the solid matrix. Must be in the calibration standard but data indicate it may not consistently produce quantitative data. See Section D.5 of Appendix DoD-D for further explanation.

TABLE C-2. SW-846 METHOD 8270C (SEMIVOLATILE ORGANIC COMPOUNDS) TARGET ANALYTE LIST (Continued)

Semivolatile Compound	CAS #	Comments	Semivolatile Compound	CAS #	Comments
2-Chloronaphthalene	91-58-7		***4-Nitrophenol***[8]	100-02-7	
2-Chlorophenol	95-57-8		N-Nitrosodimethylamine	62-75-9	
4-Chlorophenyl phenyl ether	7005-72-3		N-Nitrosodiphenylamine	86-30-6	
Chrysene	218-01-9	See also 8310	N-Nitrosodi-n-propylamine	621-64-7	
Dibenz(a,h)anthracene	53-70-3	See also 8310	N-Nitrosopyrrolidine	930-55-2	
Dibenzofuran	132-64-9		Pentachlorophenol	87-86-5	
Di-n-butyl phthalate	84-74-2		Phenanthrene	85-01-8	See also 8310
1,2-Dichlorobenzene	95-50-1	See also 8260B	***Phenol***[8]	108-95-2	
1,3-Dichlorobenzene	541-73-1	See also 8260B	Pyrene	129-00-0	See also 8310
1,4-Dichlorobenzene	106-46-7	See also 8260B	1,2,4-Trichlorobenzene	120-82-1	See also 8260B
3,3'-Dichlorobenzidine[7]	91-94-1		2,4,5-Trichlorophenol	95-95-4	
2,4-Dichlorophenol	120-83-2		2,4,6-Trichlorophenol	88-06-2	
2,6-Dichlorophenol	87-65-0		2-Fluorophenol	367-12-4	Surrogate
Diethyl phthalate	84-66-2		***Phenol-d5/d6***[8]		Surrogate
2,4-Dimethylphenol	105-67-9		Nitrobenzene-d5		Surrogate
Dimethyl phthalate	131-11-3		2-Fluorobiphenyl	321-60-8	Surrogate
4,6-Dintro-2-methylphenol	534-52-1		2,4,6-Tribromophenol	118-79-6	Surrogate
2,4-Dinitrophenol	51-28-5		Terphenyl-d14	1718-51-0	Surrogate

[8] Poor performing analyte for water. Must be in the calibration standard but data indicate it may not consistently produce quantitative data. See Section D.5 of Appendix DoD-D for further explanation.

TABLE C-3. SW-846 METHODS 8280A AND 8290 (DIOXINS/FURANS) TARGET ANALYTE LIST

Dioxin/Furan Compound	CAS #	Comments
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	8280A
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	8280A
2,3,7,8-Tetrachlorodibenzo-p-dioxin	1746-01-6	8290
1,2,3,7,8-Pentachlorodibenzo-p-dioxin	40321-76-4	8290
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin	39227-28-6	8290
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin	57653-85-7	8290
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin	19408-74-3	8290
1,2,3,4,6,7,8-Heptachlorodibenzo-p-dioxin	35822-46-9	8290
1,2,3,4,6,7,8,9-Octachlorodibenzo-p-dioxin	3268-87-9	8290
2,3,7,8-Tetrachlorodibenzofuran	51207-31-9	8290
1,2,3,7,8-Pentachlorodibenzofuran	57117-41-6	8290
2,3,4,7,8-Pentachlorodibenzofuran	57117-31-4	8290
1,2,3,4,7,8-Hexachlorodibenzofuran	70648-26-9	8290
1,2,3,6,7,8-Hexachlorodibenzofuran	57117-44-9	8290
1,2,3,7,8,9-Hexachlorodibenzofuran	72918-21-9	8290
2,3,4,6,7,8-Hexachlorodibenzofuran	60851-34-5	8290
1,2,3,4,6,7,8-Heptachlorodibenzofuran	67562-39-4	8290
1,2,3,4,7,8,9-Heptachlorodibenzofuran	55673-87-7	8290
1,2,3,4,6,7,8,9-Octachlorodibenzofurans	39001-02-0	8290
$^{13}C_{12}$-1,2,3,4-TCDD		Recovery standard
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD		Recovery standard
$^{37}C1_4$-2,3,7,8-TCDD		Cleanup standard

TABLE C-4. SW-846 METHOD 8141A (ORGANOPHOSPHORUS PESTICIDES) TARGET ANALYTE LIST

Organophosphorus Pesticide Compound	CAS #	Comments
Azinphos-methyl	86-50-1	
Bolstar (Sulprofos)	33400-43-2	
Chlorpyrifos	2921-88-2	
Coumaphos	56-72-4	
Demeton-O	298-03-3	
Demeton-S	126-75-0	
Diazinon	333-41-5	
Dichlorvos (DDVP)	62-73-7	
Disulfoton	298-04-4	
Ethoprop	13194-48-4	
Fensulfothion	115-90-2	
Fenthion	55-38-9	
Merphos	150-50-5	
Naled	300-76-5	
Parathion, methyl	298-00-0	
Phorate	298-02-2	
Ronnel	299-84-3	
Stirophos (Tetrachlorovinphos)	961-11-5	
Tokuthion (Protothiofos)	34643-46-4	
Trichloronate	327-98-0	
4-Chloro-3-nitrobenzo-trifluoride	121-17-5	Surrogate
Tributyl phosphate	126-73-8	Surrogate
Triphenyl phosphate	115-86-6	Surrogate

TABLE C-5. SW-846 METHOD 8151A (PHENOXYACID HERBICIDES) TARGET ANALYTE LIST

Phenoxyacid Herbicide Compound	CAS #	Comments
2,4-D	94-75-7	
2,4-DB	94-82-6	
2,4,5-TP (Silvex)	93-72-1	
2,4,5-T	93-76-5	
Dalapon	75-99-0	
Dicamba	1918-00-9	
Dichloroprop	120-36-5	
Dinoseb	88-85-7	
MCPA	94-74-6	
MCPP	93-65-2	
2,4-Dichlorophenylacetic acid	19719-28-9	Surrogate

TABLE C-6. SW-846 METHOD 8310 (POLYNUCLEAR AROMATIC HYDROCARBONS) TARGET ANALYTE LIST

Polynuclear Aromatic Hydrocarbon Compound	CAS #	Comments
Acenaphthene	83-32-9	See also 8270C
Acenaphthylene	208-96-8	See also 8270C
Anthracene	120-12-7	See also 8270C
Benzo(a)anthracene	56-55-3	See also 8270C
Benzo(a)pyrene	50-32-8	See also 8270C
Benzo(b)fluoranthene	205-99-2	See also 8270C
Benzo(g,h,i)perylene	191-24-2	See also 8270C
Benzo(k)fluoranthene	207-08-9	See also 8270C
Chrysene	218-01-9	See also 8270C
Dibenzo(a,h)anthracene	53-70-3	See also 8270C
Fluoranthene	206-44-0	See also 8270C
Fluorene	86-73-7	See also 8270C
Indeno(1,2,3-cd)pyrene	193-39-5	See also 8270C
Naphthalene	91-20-3	See also 8270C
Phenanthrene	85-01-8	See also 8270C
Pyrene	129-00-0	See also 8270C
Decafluorobiphenyl	434-90-2	Surrogate

TABLE C-7. SW-846 METHOD 8330 (EXPLOSIVES) TARGET ANALYTE LIST[9]

Explosive Compound	CAS #	Comments
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	
1,3,5-Trinitrobenzene	99-35-4	
1,3-Dinitrobenzene	99-65-0	
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8	
Nitrobenzene	98-95-3	See also 8260B and 8270C
2,4,6-Trinitrotoluene (TNT)	118-96-7	
4-Amino-2,6-dinitrotoluene	19406-51-0	
2-Amino-4,6-dinitrotoluene	35572-78-2	
2,4-Dinitrotoluene	121-14-2	See also 8270C
2,6-Dinitrotoluene	606-20-2	See also 8270C

[9] When surrogate compounds are not identified by the client, use an analyte from the method that is not expected to be present in the samples as the surrogate.

TABLE C-7. SW-846 METHOD 8330 (EXPLOSIVES) TARGET ANALYTE LIST (Continued)

Explosive Compound	CAS #	Comments
2-Nitrotoluene	88-72-2	
3-Nitrotoluene	99-08-1	
4-Nitrotoluene	99-99-0	

TABLE C-8. SW-846 METHOD 8081A (ORGANOCHLORINE PESTICIDES) TARGET ANALYTE LIST

Organochlorine Pesticide Compound	CAS #	Comments
Aldrin	309-00-2	
alpha-BHC	319-84-6	
beta-BHC	319-85-7	
delta-BHC	319-86-8	
gamma-BHC (Lindane)	58-89-9	
alpha-Chlordane	5103-71-9	
gamma-Chlordane	5103-74-2	
4,4'-DDD	72-54-8	
4,4'-DDE	72-55-9	
4,4'-DDT	50-29-3	
Dieldrin	60-57-1	
Endosulfan I	959-98-8	
Endosulfan II	33213-65-9	
Endosulfan sulfate	1031-07-8	
Endrin	72-20-8	
Endrin aldehyde	7421-93-4	
Endrin ketone	53494-70-5	
Heptachlor	76-44-8	
Heptachlor epoxide	1024-57-3	
Hexachlorobenzene	118-74-1	
Methoxychlor	72-43-5	
Toxaphene	8001-35-2	
4-Chloro-3-nitrobenzo-trifluoride	121-17-5	Surrogate
Tetrachloro-m-xylene (TCMX)	877-09-8	Surrogate
Decachlorobiphenyl	2051-24-3	Surrogate

TABLE C-9. SW-846 METHOD 8082 (POLYCHLORINATED BIPHENYLS) TARGET ANALYTE LIST

PCB Compound	CAS #	Comments
Aroclor 1016	12674-11-2	
Aroclor 1221	11104-28-2	
Aroclor 1232	11141-16-5	
Aroclor 1242	53469-21-9	
Aroclor 1248	12672-29-6	
Aroclor 1254	11097-69-1	
Aroclor 1260	11096-82-5	
Aroclor 1268	11100-14-4	
Aroclor 1016/1260	12674-11-2/11096-82-5	
Decachlorobiphenyl	2051-24-3	Surrogate
Tetrachloro-m-xylene (TCMX)	877-09-8	Surrogate

TABLE C-10. SW-846 METHODS 6010B, 7000A SERIES, 9010B, 9012A, AND 9056 (INORGANICS) TARGET ANALYTE LIST

Inorganic Compound	CAS #	Comments
Aluminum	7429-90-5	6010B/6020
Antimony	7440-36-0	6010B//6020/7041
Arsenic	7440-38-2	6010B/6020/7060A/7061A
Barium	7440-39-3	6010B/6020
Beryllium	7440-41-7	6010B//6020/7090
Cadmium	7440-43-9	6010B/6020/7131A
Calcium	7440-70-2	6010B
Chromium	7440-47-3	6010B/6020
Chromium, hexavalent	18540-29-9	7195/7196A/7197/7198/7199
Cobalt	7440-48-4	6010B/6020
Copper	7440-50-8	6010B/6020
Iron	7439-89-6	6010B
Lead	7439-92-1	6010B/6020/7421
Magnesium	7439-95-4	6010B
Manganese	7439-96-5	6010B/6020
Mercury	7439-97-6	7470/7471/7472
Molybdenum	7439-98-7	6010B/7481
Nickel	7440-02-0	6010B/6020
Potassium	7440-09-7	6010B
Selenium	7782-49-2	6010B/7240
Silver	7440-22-4	6010B/6020
Sodium	7440-23-5	6010B
Thallium	7440-28-0	6010B/6020/7841
Vanadium	7440-62-2	6010B/7911
Zinc	7440-66-6	6010B/6020
Cyanide	57-12-5	9010B/9012A
Bromide	24959-67-9	
Chloride	16887-00-6	
Fluoride	16984-48-8	
Nitrate	14797-55-8	
Nitrite	14979-65-0	
Phosphate	14265-44-2	
Sulfate	14808-79-8	

APPENDIX DOD-D – LCS CONTROL LIMITS

DoD conducted a study to establish control limits for laboratory control samples using data collected from environmental laboratories that analyze samples for DoD. LCS recoveries for all the analytes on the target analyte lists were pooled, and statistical analyses (such as outlier tests and analysis of variance) were performed on the data before generating the final LCS control limits (LCS-CL). A complete description of the methodology and findings for method 8270C can be found in the Laboratory Control Sample Pilot Study (DoD, 2000).

Environmental testing laboratories that perform work for DoD must utilize the DoD-specified LCS control limits when assessing batch acceptance whenever they are available. This appendix presents the control limits generated by the LCS study and the methodology for applying the limits to LCS data. All analytes spiked in the LCS shall meet the DoD-generated LCS control limits. DoD will allow a number of sporadic marginal exceedances. Depending on the length of the list of analytes, a specified small number of analytes may exceed the generated control limit. These are based on a probability of 0.9 that any given analyte will exceed its LCS-CL and a probability of 9 out of 100 that the total number of exceedances for a LCS is less than the allowable value. Upper and lower marginal exceedance (ME) limits, calculated at 4 standard deviations around the mean, are established to mark the boundaries of marginal exceedances. If more analytes exceed the LCS-CLs than is allowed, or if any one analyte exceeds the ME limits, then the LCS has failed. This marginal exceedance approach is relevant for methods with longer lists of analytes. It will not apply to some target analyte lists (fewer than 30 analytes).

DoD LCS Control Limits Policy

- The laboratory shall use project-specific control limits based on data quality objectives (DQOs), if available. If not, DoD-generated LCS-CLs shall be used, if available. Otherwise, the lab's own in-house control limits shall be used.
- The LCS-CLs are based on the current promulgated versions of SW-846 methods at the time of the study (2000). They should be used as a benchmark to evaluate acceptability even as methods are updated or alternative methods for the same class of compounds become available.
- The fact that the LCS-CLs are based on certain SW-846 methods should not limit the use of alternative analytical methods, as appropriate. If an alternative method is used, however, it should be capable of producing LCS recoveries that are at least as good as the DoD-generated LCS-CLs, unless project-specific DQOs allow less stringent criteria.
- The LCS study shows that preparatory methods may have a significant influence on a laboratory's ability to achieve certain LCS-CLs. If a laboratory is unable to achieve the LCS-CLs presented in this appendix, it should investigate the use of alternative preparatory methods as a means to improve precision and accuracy.

D.1 Generated LCS Control Limits

As mentioned above, DoD compiled LCS data from multiple laboratories, performing statistical analyses on the data sets before generating control limits. The control limits were set at 3 standard deviations around the mean for all methods except 8151A (see below for further explanation). The ME limits were set at 4 standard deviations around the mean. The lower ME limit was then raised to 10% for those analytes in which 4 standard deviations falls below that level. Tables D-4 through D-7 and D-10 through D-19 at the end of this appendix present the mean, standard deviation, lower control limit, and upper control limit for each analyte in methods 8260B, 8270C, 8310, 8330, 8081A, 8082, 6010, and 7470A/7471A, for the water and solid matrices. The lower and upper ME limits are presented for methods 8260B and 8270C as well, since those are the two methods with greater than 30 analytes and therefore capable of utilizing the sporadic marginal exceedance allowance. The analytes for method 8270C are grouped by compound class.

The control limits for explosives method 8330 in the water matrix were generated using data that were based on solid phase extraction (SPE) with acetonitrile elution (method 3535A) only. Analysis of the data received from the LCS study showed that the SPE method produced recoveries with higher means and lower standard deviations than the salting out extraction method. This results in significantly narrower control limits. Since SPE produces higher recoveries and is less expensive, cumbersome and time and labor intensive, the LCS control limits for method 8330 in water were set with data using only that method. A limited amount of data were received that used SPE/acetonitrile, therefore, no outliers were removed during the statistical analysis. This ensures a representative data set was used to generate the control limits (see Table D-12). Note: Laboratories may use any extraction method they feel is appropriate; however, the LCS recoveries must fall within the LCS-CLS presented in Table D-12.

Control limits for phenoxyacid herbicides method 8151A were generated using a non-parametric statistical approach. This is a different approach than for the other methods in the LCS study due to the large amount of intralaboratory variability in recoveries for all analytes in the method. The control limits for method 8151A, both solid and water matrices, were set at the 5^{th} and 95^{th} percentile of all data received in the study (no outliers were removed). Tables D-8 and D-9 present the median, lower control limit, and upper control limit for each analyte. LCS failure is assessed and corrective action applied the same way for all methods with control limits in this appendix (see Sections D.3 and D.4).

[Note: These data represent the current capability of the SW-846 analytical and preparatory methods. Use of alternative preparatory procedures and/or improvements through PBMS are encouraged. Project-specific control limits can supersede these DoD limits.] If limits are not available for a project-specific analyte, the laboratory shall discuss with the client appropriate limits considering the project-specific DQOs.

D.2 Marginal Exceedance

DoD will allow a statistically based number of sporadic marginal exceedances of the LCS-CLs. The number of exceedances is based on the total number of analytes spiked in the LCS. As the number of analytes in the LCS increases, more marginal exceedances are allowed. The number of allowable marginal exceedances is based on a probability of 0.9 that any given analyte will exceed its LCS-CL and a probability of 9 out of 100 that the total number of exceedances for a LCS is outside the allowable value. Table D-1 presents the allowable number of marginal exceedances for a given number of analytes in the LCS.

TABLE D-1. NUMBER OF MARGINAL EXCEEDANCES

Number of Analytes in LCS	Allowable Number of Marginal Exceedances of LCS-CLs
74 – 70	5
69 – 60	4
59 – 51	3
50 – 40	2
39 – 30	1
< 30	0

A *marginal* exceedance is defined as beyond the LCS-CL but still within the marginal exceedance limits (set at 4 standard deviations around the mean, see Tables D-4 through D-7 for method 8260B and 8270C). This outside boundary prevents a grossly out-of-control LCS from passing.

DoD requires that the marginal exceedances be sporadic (i.e., random). If the same analyte exceeds the LCS-CL repeatedly, that is an indication that the problem is systemic and something is wrong with the measurement system. The source of error should be located and the appropriate corrective action taken. Laboratories must monitor through QA channels the application of the sporadic marginal exceedance allowance to the LCS results to ensure random behavior. The allowance for marginal exceedances is a new policy being introduced DoD-wide. Its effective implementation requires cooperation from the laboratory. If the laboratory fails to implement the policy properly, the privilege of using the marginal exceedance option will be revoked. Oversight and appropriate corrective action will be a focus of DoD laboratory audits in the future.

D.3 LCS Failure

Each LCS must be evaluated against DoD's control limits and ME limits before being accepted (see Tables D-4 through D-19). First, the recoveries for the analytes spiked in the LCS should be compared to the LCS control limits. If a recovery is less than the lower control limit or greater than the upper control limit, that is an exceedance. The laboratory should note which analytes exceeded the control limits and make a comparison to the list of project-specific analytes of concern. **If a project-specific analyte of concern exceeds its LCS-CL, the LCS has failed.** Next, the laboratory should add up the total number of exceedances for the LCS. Based on the number of analytes spiked in the LCS, the total number of exceedances should be compared with the allowable number from Table D-1. **If a LCS has more than the allowable number of marginal exceedances, the LCS has failed.** Finally, the recoveries for those analytes that exceeded the LCS-CL should be compared to the ME limits from Tables D-4 through D-7. **If a single analyte exceeds its marginal exceedance limit, the LCS has failed.** (This applies only to methods 8260B and 8270C because of the large size of the analyte lists.)

Note: The target analytes from Appendix DoD-C should not be considered project-specific analytes of concern unless the client separately specifies the analytes. A requirement to analyze all compounds on the target analyte list does not define a project-specific analyte.

In summary, failure of the LCS can occur several ways:

- Exceedance of a LCS-CL by any project-specific analyte of concern
- Marginal exceedance of the LCS-CLs by more than the allowable number of analytes
- Exceedance of the ME limits by one or more analytes

Once a LCS has failed, corrective action is required (see section D.4).

D.4 Corrective Action

If a sample fails based on any of the criteria in section D.3, corrective action is required. The corrective action requirement applies to all analytes that exceeded the LCS-CLs, even if one specific analyte's exceedance was not the trigger of LCS failure (see example in text box). **All exceedances of the LCS-CLs, marginal or otherwise, are subject to corrective action.**

If a LCS fails, an attempt must be made to determine the source of error and find a solution. All the findings and corrective action should be documented. DoD then requires that the analytes subject to corrective action in the LCS and all the samples in the batch be reprepped and reanalyzed or the batch rerun with a new LCS. The corrective action applied shall be based on professional judgment in the review of other QC measures (i.e., surrogates). If an analyte falls outside the LCS-CL a second time or if there is not sufficient sample material available to be reanalyzed, then all the results in the associated batch for that analyte must be flagged with a "Q" (see DoD clarification box D-19). The recoveries of those analytes subject to corrective action must be documented in the case narrative, whether flagging is needed or not.

Example of Applying Corrective Action

- In a single LCS, anthracene has a recovery of 30%.
- The lower ME limit for anthracene is 44, therefore the LCS has failed.
- In the same LCS three other analytes exceeded their LCS-CLs but were within their ME limits.
- The LCS was spiked with 74 analytes; therefore, according to Table D-1, five marginal exceedances are allowed.
- The four total exceedances (anthracene plus the three other analytes) are within the allowable number for that analyte list size.

Corrective action is triggered for the LCS because the anthracene recovery exceeded its ME limit, but it is required for all four analytes that exceeded the LCS-CLs.

D.5 Poor Performing Analytes

On the basis of results from the LCS study, DoD identified certain compounds that do not perform well with specific methods. These compounds produce low mean recoveries and high standard deviations, resulting in wide LCS control limits with particularly low lower control limits (sometimes negative values). The performance of these compounds reflects routine implementation of the method in many laboratories. DoD has defined a poor performing analyte as having a lower control limit of 10% or less. DoD does not feel it is appropriate to control batch acceptance on these compounds because there is a high level of uncertainty in their recovery. The data may be used; however, routine performance of the method on these compounds can result in being able to identify only a small percentage of the analyte.

The laboratory should include all target analytes in the calibration standard, including the poor performing analytes. If one of the poor performing analytes identified below is a project-specific analyte of concern *or* if it is detected in the project samples, the laboratory should contact the client (DoD), who will then work with the laboratory on an appropriate course of action. Ideally DoD and the laboratory would use an alternative method to test for the analyte (one that is known to produce higher recoveries) or else modify the original method to optimize conditions for the poor performing analyte.

Poor performing analytes were only identified in SW-846 methods 8270C, 8151A, and 8330. These analytes, along with the mean, standard deviation, lower control limit, upper control limit, lower ME limit, and upper ME limit (as generated by the LCS study) are presented in Table D-2. [Note: Lower limits calculated as negative values were raised to zero.]

TABLE D-2. POOR PERFORMING ANALYTES[10]

Analyte	Mean/ Median	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
8270C Water:						
4-Nitrophenol	54	23	0	123	0	146
Benzoic acid	54	24	0	127	0	151
Phenol	55	19	0	116	0	136
Phenol-d5/d6 (surrogate)	62	18	9	117	0	135
8270C Solid:						
3,3'Dichlorobenzidine	68	19	10	128	0	147
4-Chloroaniline	51	14	8	94	0	108
Benzoic acid	55	18	0	112	0	130
8151A Solid:						
Dinoseb	72		5	131		
8330 Solid:						
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	80	23	10	150		

The LCS control limits generated by the study for the poor performing analytes are provided as a benchmark against which laboratories may measure the effectiveness of modifications to the current methods. Batch acceptance should not be calculated using these limits. Laboratories should attempt to raise the mean recoveries and lower the standard deviations. No recovery of less than 10% will be considered acceptable, however.

D.6 Surrogates

The surrogate compounds for each method are added to all samples, standards, and blanks to assess the ability of the method to recover specific non-target analytes from a given matrix and to monitor sample-specific recovery. Control limits for these compounds were calculated in the same study as the other analytes on the target analyte

[10] Control limits for method 8151A were generated using non-parametric statistics; therefore, the median and no standard deviation is presented (see Section D.1 for further explanation). ME limits are not used for methods 8151A and 8330 since the target analyte lists have less than 30 analytes.

lists. Below are the limits for some of the surrogates of methods 8260B, 8270C, 8081A, and 8082, based on 3 standard deviations around the mean (Table D-3). Control limits are not available for some surrogates that appear on the target analyte lists in Appendix DoD-C. Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. No ME limits are presented as marginal exceedances are not acceptable for surrogate spikes. Note: DoD prefers the use of those surrogates not identified as poor performing analytes in Table D-2 above.

TABLE D-3. SURROGATES

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
8260B Water:				
1,2-Dichloroethane-d4	95	8	72	119
4-Bromofluorobenzene	98	7	76	119
Dibromofluoromethane	100	5	85	115
Toluene-d8	102	6	83	120
8260B Solid:				
4-Bromofluorobenzene	101	6	84	118
Toluene-d8	100	5	84	116
8270C Water:				
2-Fluorobiphenyl	79	10	48	112
Terphenyl-d14	92	14	51	135
2,4,6-Tribromophenol	82	13	42	124
2-Fluorophenol	63	14	19	108
Nitrobenzene-d5	76	11	41	111
8270C Solid:				
2-Fluorobiphenyl	72	10	43	103
Terphenyl-d14	78	15	32	125
2,4,6-Tribromophenol	80	15	36	126
2-Fluorophenol	70	11	37	104
Phenol-d5/d6	71	10	40	102
Nitrobenzene-d5	69	10	37	102
8081A Water:				
Decachlorobiphenyl	83	17	32	135
TCMX	81	19	25	138
8081A Solid:				
Decachlorobiphenyl	94	13	56	132
TCMX	97	9	69	124
8082 Water:				
Decachlorobiphenyl	88	15	42	133
8082 Solid:				
Decachlorobiphenyl	91	11	58	125

D.7 In-House LCS Limits

SW-846 methods recommend that laboratories calculate LCS limits on a semiannual basis using at least 15 to 20 data points. The existence of DoD-wide LCS control limits should not eliminate the practice of laboratories' generating independent in-house LCS limits based on statistical analysis of historical LCS results. Laboratories must continue to generate in-house LCS limits annually for all analytes. These limits shall be compared to the DoD LCS control limits, where available, and to the laboratory's in-house limits from the previous year. The in-house limits shall be used as a quality control measure to evaluate change in the laboratory's performance over time. The acceptability of LCS results within any preparatory batch shall no longer be based on the in-house limits, unless DoD has not published LCS-CLs for a particular analyte.

In addition, DoD strongly recommends that the laboratory track trends in the LCS data over time through the use of control charts. This QA measure can identify potential problems within the measurement system before they result in method failure. The control charts should be reviewed by the quality assurance officer or designee on a regular basis and corrective action implemented when necessary.

TABLE D-4. LCS CONTROL LIMITS FOR SW-846 METHOD 8260B WATER MATRIX[11]

Analyte	Mean	Standard Deviation	Lower Control Limit[12]	Upper Control Limit[12]	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	105	8	81	129	73	137
1,1,1-Trichloroethane	100	11	67	132	56	143
1,1,2,2-Tetrachloroethane	96	11	63	128	53	138
1,1,2-Trichloroethane	100	8	75	125	66	133
1,1-Dichloroethane	101	11	69	133	58	143
1,1-Dichloroethene	99	10	68	130	57	140
1,1-Dichloropropene	102	10	73	132	63	142
1,2,3-Trichlorobenzene	99	14	57	142	43	156
1,2,3-Trichloropropane	98	9	73	124	64	132
1,2,4-Trichlorobenzene	100	11	66	134	54	145
1,2,4-Trimethylbenzene	103	10	74	132	64	142
1,2-Dibromo-3-chloropropane	91	14	50	132	37	146
1,2-Dibromoethane	100	7	80	121	73	127
1,2-Dichlorobenzene	96	9	71	122	62	131
1,2-Dichloroethane	100	10	69	132	58	142
1,2-Dichloropropane	100	8	75	125	67	134
1,3,5-Trimethylbenzene	102	10	74	131	64	140
1,3-Dichlorobenzene	100	8	75	124	67	132
1,3-Dichloropropane	100	9	73	126	64	135
1,4-Dichlorobenzene	99	8	74	123	66	131
2,2-Dichloropropane	103	11	69	137	58	148
2-Butanone	91	20	32	150	12	170
2-Chlorotoluene	100	9	73	126	64	135
2-Hexanone	92	12	56	128	44	140
4-Chlorotoluene	101	9	74	128	65	137
4-Methyl-2-pentanone	96	13	58	134	45	147
Acetone	91	17	39	142	22	159
Benzene	102	7	81	122	74	129
Bromobenzene	100	8	76	124	69	131
Bromochloromethane	97	11	65	129	55	140
Bromodichloromethane	98	8	76	121	68	128
Bromoform	99	10	69	128	59	138
Bromomethane	88	19	30	146	10	166
Carbon disulfide	100	21	37	162	17	183
Carbon tetrachloride	102	12	66	138	54	150
Chlorobenzene	102	7	81	122	74	129
Chlorodibromomethane	96	13	58	133	46	146
Chloroethane	99	12	62	135	50	147
Chloroform	100	12	63	136	51	149

[11] LCS control limits are not available for Total Xylene because although Xylene may be reported on a project-specific basis as a total number. For the purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section D.5 and for surrogate compounds in section D.6.

[12] Only applied to the acceptable number of marginal exceedances based on the analyte list size from Table D-1.

TABLE D-4. LCS CONTROL LIMITS FOR SW-846 METHOD 8260B WATER MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit[12]	Upper Control Limit[12]	Lower ME Limit	Upper ME Limit
Chloromethane	83	15	39	127	25	142
cis-1,2-Dichloroethene	99	9	72	126	62	135
cis-1,3-Dichloropropene	100	10	69	131	59	142
Dibromomethane	101	8	76	125	67	134
Dichlorodifluoromethane	93	21	31	155	11	175
Ethylbenzene	100	9	73	127	64	137
Hexachlorobutadiene	97	15	51	142	36	158
Isopropylbenzene	101	9	75	127	66	136
m,p-Xylene	102	9	76	128	67	137
Methyl tert-butyl ether	94	10	65	123	55	133
Methylene chloride	96	14	53	140	39	154
Naphthalene	96	14	54	138	40	152
n-Butylbenzene	103	11	69	137	57	148
n-Propylbenzene	101	9	72	129	63	138
o-Xylene	100	7	80	121	73	128
p-Isopropyltoluene	102	10	73	131	63	141
sec-Butylbenzene	100	9	72	127	63	137
Styrene	100	11	65	134	54	146
tert-Butylbenzene	99	10	70	129	60	139
Tetrachloroethene	96	18	44	149	26	167
Toluene	100	7	77	122	70	130
trans-1,2-Dichloroethene	99	13	60	139	46	152
trans-1,3-Dichloropropene	98	15	53	142	39	157
Trichloroethene	99	9	70	127	61	136
Trichlorofluoromethane	103	15	59	146	44	161
Vinyl chloride	99	16	50	147	34	163

TABLE D-5. LCS CONTROL LIMITS FOR SW-846 METHOD 8260B SOLID MATRIX[13]

Analyte	Mean	Standard Deviation	Lower Control Limit[14]	Upper Control Limit[14]	Lower ME Limit	Upper ME Limit
1,1,1,2-Tetrachloroethane	100	9	74	125	65	134
1,1,1-Trichloroethane	101	11	68	133	57	144
1,1,2,2-Tetrachloroethane	93	13	54	131	41	144
1,1,2-Trichloroethane	95	11	62	127	51	138
1,1-Dichloroethane	99	9	73	125	64	134
1,1-Dichloroethene	100	12	65	136	53	148
1,1-Dichloropropene	102	11	70	135	59	145
1,2,3-Trichlorobenzene	97	12	62	133	51	144
1,2,3-Trichloropropane	97	11	63	130	52	141
1,2,4-Trichlorobenzene	98	11	65	131	54	142
1,2,4-Trimethylbenzene	100	12	65	135	53	147
1,2-Dibromo-3-chloropropane	87	16	40	135	25	150

[13] LCS control limits are not available for Methyl tert-butyl ether and Total Xylene although those compounds do appear on the target analyte list for method 8260B (Table C-1 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for MTBE during the LCS study. Xylene may be reported on a project-specific basis as a total number; however, for the purposes of the DoD QSM, it will be analyzed and reported as m,p-Xylene and o-Xylene. Additional limits for poor performing compounds can be found in section D.5 and for surrogate compounds in sectionD.6.
[14] Only applied to the acceptable number of marginal exceedances based on the analyte list size from Table D-1.

TABLE D-5. LCS CONTROL LIMITS FOR SW-846 METHOD 8260B SOLID MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit[14]	Upper Control Limit[14]	Lower ME Limit	Upper ME Limit
1,2-Dibromoethane	97	9	70	124	61	133
1,2-Dichlorobenzene	97	7	74	119	67	126
1,2-Dichloroethane	104	11	72	137	61	147
1,2-Dichloropropane	95	8	71	119	63	127
1,3,5-Trimethylbenzene	99	11	65	133	54	144
1,3-Dichlorobenzene	98	9	72	124	63	133
1,3-Dichloropropane	100	8	76	123	68	131
1,4-Dichlorobenzene	98	9	72	125	63	134
2,2-Dichloropropane	101	11	67	134	55	146
2-Butanone	94	22	29	159	10	180
2-Chlorotoluene	98	10	69	128	59	138
2-Hexanone	97	16	47	146	31	162
4-Chlorotoluene	100	9	73	126	65	135
4-Methyl-2-pentanone	97	17	47	147	31	164
Acetone	88	23	19	158	10	181
Benzene	99	9	73	126	64	134
Bromobenzene[15]	93	9	66	121	56	130
Bromochloromethane	99	9	71	127	62	137
Bromodichloromethane	100	9	72	128	62	137
Bromoform	96	13	56	137	43	150
Bromomethane	95	21	31	159	10	180
Carbon disulfide	103	19	47	159	28	178
Carbon tetrachloride	100	11	67	133	56	144
Chlorobenzene	99	8	75	123	67	131
Chlorodibromomethane	98	11	66	130	56	140
Chloroethane	98	20	39	157	20	177
Chloroform	98	9	72	124	63	133
Chloromethane	90	13	51	129	38	142
cis-1,2-Dichloroethene	96	10	67	125	57	135
cis-1,3-Dichloropropene	99	9	72	126	63	135
Dibromomethane	100	9	73	128	63	137
Dichlorodifluoromethane[15]	85	17	34	136	17	153
Ethylbenzene	101	9	74	127	65	136
Hexachlorobutadiene	98	15	53	142	38	157
Isopropylbenzene	103	9	77	129	68	138
m,p-Xylene	102	8	79	126	71	134
Methylene chloride	97	14	54	141	40	155
Naphthalene	84	14	40	127	26	141
n-Butylbenzene	101	12	65	138	52	150
n-Propylbenzene	99	12	63	135	51	147
o-Xylene	101	8	77	125	69	133
p-Isopropyltoluene	104	10	75	133	65	142
sec-Butylbenzene	97	11	63	132	51	143
Styrene	101	9	74	128	65	137
tert-Butylbenzene	99	11	65	132	54	143
Tetrachloroethene	103	12	67	139	55	150
Toluene	99	9	71	127	62	136
trans-1,2-Dichloroethene	100	11	66	134	55	145
trans-1,3-Dichloropropene	96	10	65	127	54	138

[15] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from less than four laboratories. Limits may be adjusted in the future as additional data becomes available.

TABLE D-5. LCS CONTROL LIMITS FOR SW-846 METHOD 8260B SOLID MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit[14]	Upper Control Limit[14]	Lower ME Limit	Upper ME Limit
Trichloroethene	101	8	77	124	69	132
Trichlorofluoromethane	106	27	25	186	10	213
Vinyl chloride	92	11	58	126	46	138

TABLE D-6. LCS CONTROL LIMITS FOR SW-846 METHOD 8270C WATER MATRIX[16]

Analyte	Mean	Standard Deviation	Lower Control Limit[17]	Upper Control Limit[17]	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	75.0	9.5	46	104	37	113
Acenaphthene	77.6	10.1	47	108	37	118
Acenaphthylene	78.5	9.4	50	107	41	116
Anthracene	83.0	9.7	54	112	44	122
Benz(a)anthracene	82.7	8.9	56	109	47	118
Benzo(a)pyrene	81.3	9.5	53	110	43	119
Benzo(b)fluoranthene	81.8	12.1	45	118	33	130
Benzo(g,h,i)perylene	80.5	14.1	38	123	24	137
Benzo(k)fluoranthene	84.6	13.2	45	124	32	137
Chrysene	82.1	8.9	55	109	46	118
Dibenz(a,h)anthracene	84.7	14.1	42	127	28	141
Fluoranthene	85.2	10.4	54	116	44	127
Fluorene	80.6	10.3	50	112	39	122
Indeno(1,2,3-cd)pyrene	84.3	13.6	43	125	30	139
Naphthalene	70.8	10.5	39	102	29	113
Phenanthrene	84.0	11.0	51	117	40	128
Pyrene	88.6	13.2	49	128	36	142
Phenolic/Acidic						
2,4,5-Trichlorophenol	79.7	10.3	49	111	38	121
2,4,6-Trichlorophenol	80.7	10.7	49	113	38	123
2,4-Dichlorophenol	76.3	9.6	48	105	38	115
2,4-Dimethylphenol	68.8	13.5	28	109	15	123
2,4-Dinitrophenol	75.8	20.6	14	138	10	158
2-Chlorophenol	71.3	11.4	37	106	26	117
2-Methylphenol	73.3	11.7	38	109	26	120
2-Nitrophenol	75.8	12.4	39	113	26	125
3-Methylphenol/4-Methylphenol	71.3	13.0	32	110	19	123
4,6-Dinitro-2-methylphenol	84.9	15.0	40	130	25	145

[16] LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270C (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

[17] Only applied to the acceptable number of marginal exceedances based on the analyte list size from Table D-1.

TABLE D-6. LCS CONTROL LIMITS FOR SW-846 METHOD 8270C WATER MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit[18]	Upper Control Limit[18]	Lower ME Limit	Upper ME Limit
4-Chloro-3-methylphenol	78.6	10.7	47	111	36	121
Pentachlorophenol	77.6	13.3	38	117	24	131
Basic						
3,3'-Dichlorobenzidine	65.2	15.3	19	111	10	126
4-Chloroaniline	62.2	15.6	15	109	10	125
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	84.2	14.0	42	126	28	140
Butyl benzyl phthalate	81.1	11.7	46	116	34	128
Di-n-butyl phthalate	84.8	10.3	54	116	44	126
Di-n-octyl phthalate	87.4	16.6	37	137	21	154
Diethyl phthalate	79.2	12.9	41	118	28	131
Dimethyl phthalate	75.9	16.9	25	127	10	144
Nitrosoamines						
N-Nitrosodi-n-propylamine	80.9	15.7	34	128	18	144
N-Nitrosodimethylamine	67.9	14.1	26	110	11	124
N-Nitrosodiphenylamine	79.6	10.6	48	111	37	122
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	76.2	10.2	46	107	35	117
Bis(2-chloroethyl) ether	73.3	12.3	37	110	24	122
Bis(2-chloroisopropyl) ether	78.2	17.5	26	131	10	148
Hexachlorobutadiene	65.2	12.6	27	103	15	116
Hexachloroethane	60.9	11.1	28	94	16	105
Halogenated Aromatics						
1,2,4-Trichlorobenzene	71.7	11.6	37	107	25	118
1,2-Dichlorobenzene	67.3	11.4	33	102	22	113
1,3-Dichlorobenzene	64.8	10.9	32	98	21	108
1,4-Dichlorobenzene	64.8	10.9	32	98	21	109
2-Chloronaphthalene	76.5	9.3	49	104	39	114
4-Bromophenyl phenyl ether	82.9	10.2	52	113	42	124
4-Chlorophenyl phenyl ether	80.6	10.3	50	111	39	122
Hexachlorobenzene	82.3	10.0	52	112	42	122
Nitroaromatics						
2,4-Dinitrotoluene	84.3	11.2	51	118	39	129
2,6-Dinitrotoluene	82.7	11.3	49	117	37	128
2-Nitroaniline	81.8	11.2	48	115	37	126
3-Nitroaniline	72.6	17.7	19	126	10	143
4-Nitroaniline	77.2	13.7	36	118	22	132
Nitrobenzene	76.8	10.8	44	109	34	120
Neutral Aromatics						
Carbazole	82.5	11.4	48	117	37	128
Dibenzofuran	80.3	8.8	54	107	45	115

[18] Only applied to the acceptable number of marginal exceedances based on the analyte list size from Table D-1.

TABLE D-6. LCS CONTROL LIMITS FOR SW-846 METHOD 8270C WATER MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit[19]	Upper Control Limit[19]	Lower ME Limit	Upper ME Limit
Others						
1,2-Diphenylhydrazine	84.8	9.4	57	113	47	122
Benzyl alcohol	71.0	13.8	30	112	16	126
Isophorone	81.0	10.5	50	112	39	123

TABLE D-7. LCS CONTROL LIMITS FOR SW-846 METHOD 8270C SOLID MATRIX[20]

Analyte	Mean	Standard Deviation	Lower Control Limit[21]	Upper Control Limit[21]	Lower ME Limit	Upper ME Limit
Polynuclear Aromatics						
2-Methylnaphthalene	77.3	10.0	47	107	37	117
Acenaphthene	77.3	10.3	46	108	36	119
Acenaphthylene	75.7	10.4	44	107	34	117
Anthracene	79.9	9.0	53	107	44	116
Benz(a)anthracene	81.6	9.8	52	111	42	121
Benzo(a)pyrene	80.7	10.3	50	111	40	122
Benzo(b)fluoranthene	79.7	11.4	45	114	34	125
Benzo(g,h,i)perylene	81.8	14.7	38	126	23	140
Benzo(k)fluoranthene	83.8	12.9	45	123	32	136
Chrysene	82.6	9.9	53	112	43	122
Dibenz(a,h)anthracene	82.9	13.9	41	125	27	139
Fluoranthene	83.9	10.1	54	114	44	124
Fluorene	78.3	9.8	49	108	39	117
Indeno(1,2,3-cd)pyrene	79.7	13.8	38	121	25	135
Naphthalene	73.4	11.1	40	107	29	118
Phenanthrene	80.1	10.0	50	110	40	120
Pyrene	84.4	12.8	46	123	33	136
Phenolic/Acidic						
2,4,5-Trichlorophenol	80.1	10.4	49	111	38	122
2,4,6-Trichlorophenol	76.3	11.0	43	109	32	120
2,4-Dichlorophenol	77.2	10.9	45	110	34	121
2,4-Dimethylphenol	67.3	11.9	32	103	20	115
2,4-Dinitrophenol	72.6	20.0	13	132	10	152
2-Chlorophenol	74.7	10.3	44	106	34	116
2-Methylphenol	71.7	10.6	40	104	29	114
2-Nitrophenol	76.2	11.5	42	111	30	122

[19] Only applied to the acceptable number of marginal exceedances based on the analyte list size from Table D-1.
[20] LCS control limits are not available for Benzidine, 2,6-Dichlorophenol, 1,2-Diphenylhydrazine, and N-nitrosopyrrolidine, although those compounds do appear on the target analyte list for method 8270C (Table C-2 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.
[21] Only applied to the acceptable number of marginal exceedances based on the analyte list size from Table D-1.

TABLE D-7. LCS CONTROL LIMITS FOR SW-846 METHOD 8270C SOLID MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit[22]	Upper Control Limit[22]	Lower ME Limit	Upper ME Limit
3-Methylphenol/4-Methylphenol	73.9	10.9	41	107	30	118
4,6-Dinitro-2-methylphenol	83.1	18.0	29	137	11	155
4-Chloro-3-methylphenol	79.5	11.1	46	113	35	124
4-Nitrophenol	77.0	20.2	17	138	10	158
Pentachlorophenol	71.9	15.6	25	119	10	134
Phenol	69.7	10.2	39	100	29	110
Phthalate Esters						
Bis(2-ethylhexyl) phthalate	87.4	13.3	47	127	34	141
Butyl benzyl phthalate	86.4	12.3	49	123	37	136
Di-n-butyl phthalate	83.2	9.1	56	110	47	120
Di-n-octyl phthalate	86.4	15.2	41	132	26	147
Diethyl phthalate	82.2	10.6	50	114	40	125
Dimethyl phthalate	79.6	10.2	49	110	39	120
Nitrosoamines						
N-Nitrosodi-n-propylamine	76.8	12.3	40	114	28	126
N-Nitrosodimethylamine	66.1	15.9	18	114	10	129
N-Nitrosodiphenylamine	82.4	11.1	49	116	38	127
Chlorinated Aliphatics						
Bis(2-chlorethoxy)methane	75.5	10.9	43	108	32	119
Bis(2-chloroethyl) ether	71.1	11.2	38	105	26	116
Bis(2-chloroisopropyl) ether	68.4	15.7	21	115	10	131
Hexachlorobutadiene	78.2	12.9	40	117	27	130
Hexachloroethane	71.9	12.6	34	110	22	122
Halogenated Aromatics						
1,2,4-Trichlorobenzene	77.4	11.2	44	111	32	122
1,2-Dichlorobenzene	70.9	8.7	45	97	36	106
1,3-Dichlorobenzene	69.7	10.3	39	100	29	111
1,4-Dichlorobenzene	69.0	11.4	35	103	23	115
2-Chloronaphthalene	75.2	9.9	45	105	35	115
4-Bromophenyl phenyl ether	81.7	11.8	46	117	34	129
4-Chlorophenyl phenyl ether	79.6	10.7	47	112	37	122
Hexachlorobenzene	82.5	11.7	47	118	36	129
Nitroaromatics						
2,4-Dinitrotoluene	82.0	11.4	48	116	36	128
2,6-Dinitrotoluene	80.2	10.7	48	112	37	123
2-Nitroaniline	81.0	12.2	44	118	32	130
3-Nitroaniline	68.8	13.8	27	110	13	124
4-Nitroaniline	73.6	13.1	34	113	21	126
Nitrobenzene	77.2	11.9	41	113	30	125
Neutral Aromatics						
Carbazole	80.4	12.3	44	117	31	129
Dibenzofuran	77.1	8.8	51	103	42	112

[22] Only applied to the acceptable number of marginal exceedances based on the analyte list size from Table D-1.

TABLE D-7. LCS CONTROL LIMITS FOR SW-846 METHOD 8270C SOLID MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit[23]	Upper Control Limit[23]	Lower ME Limit	Upper ME Limit
Others						
Benzyl alcohol	70.9	17.4	19	123	10	140
Isophorone	77.0	11.4	43	111	31	123

TABLE D-8. LCS CONTROL LIMITS FOR SW-846 METHOD 8151A WATER MATRIX[24]

Analyte	Median	Lower Control Limit	Upper Control Limit
2,4-D	88	35	113
2,4-DB	99	44	132
2,4,5-T	83	34	112
2,4,5-TP (Silvex)	87	49	116
Dalapon	62	40	108
Dicamba	86	60	112
Dichloroprop	91	68	122
Dinoseb	65	21	97
MCPA	93	62	144

TABLE D-9. LCS CONTROL LIMITS FOR SW-846 METHOD 8151A SOLID MATRIX[25]

Analyte	Median	Lower Control Limit	Upper Control Limit
2,4-D	88	36	144
2,4-DB	108	52	157
2,4,5-T	86	43	137
2,4,5-TP (Silvex)	90	46	125
Dicamba	90	56	110
Dichloroprop	99	77	138

[23] Only applied to the acceptable number of marginal exceedances based on the analyte list size from Table D-1.

[24] LCS control limits were generated using non-parametric statistics (see section D.1 for further explanation). LCS control limits are not available for MCPP, although the compound does appear on the target analyte list for method 8151A (Table C-5 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for the analyte during the LCS study.

[25] LCS control limits were generated using non-parametric statistics (see section D.1 for further explanation). LCS control limits are not available for Dalapon, MCPA, and MCPP, although those compounds do appear on the target analyte list for method 8151A (Table C-5 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for poor performing compounds can be found in section D.5.

TABLE D-10. LCS CONTROL LIMITS FOR SW-846 METHOD 8310 WATER MATRIX

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Acenaphthene	70	11	35	104
Acenaphthylene	74	13	34	113
Anthracene	77	12	41	112
Benzo(a)anthracene	81	11	49	112
Benzo(a)pyrene	79	11	45	113
Benzo(b)fluoranthene	82	10	51	112
Benzo(g,h,i)perylene	77	14	34	119
Benzo(k)fluoranthene	79	10	48	110
Chrysene	83	11	50	116
Dibenzo(a,h)anthracene	64	15	18	111
Fluoranthene	82	11	48	116
Fluorene	69	11	35	103
Indeno(1,2,3-cd)pyrene	80	11	47	112
Naphthalene	68	12	33	104
Phenanthrene	80	13	40	120
Pyrene	80	9	52	108

TABLE D-11. LCS CONTROL LIMITS FOR SW-846 METHOD 8310 SOLID MATRIX

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Acenaphthene	71	12	33	108
Acenaphthylene	73	13	33	113
Anthracene	86	13	47	125
Benzo(a)anthracene	78	9	50	106
Benzo(a)pyrene	86	15	40	133
Benzo(b)fluoranthene	89	11	57	121
Benzo(g,h,i)perylene[26]	85	10	53	116
Benzo(k)fluoranthene	84	12	48	121
Chrysene	87	11	55	119
Dibenzo(a,h)anthracene	81	11	47	115
Fluoranthene	88	16	41	135
Fluorene	76	10	46	107
Indeno(1,2,3-cd)pyrene	95	13	56	134
Naphthalene	80	11	48	111
Phenanthrene	91	12	57	126
Pyrene	82	11	49	115

[26] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from less than four laboratories. Limits may be adjusted in the future as additional data becomes available.

TABLE D-12. LCS CONTROL LIMITS FOR SW-846 METHOD 8330 WATER MATRIX[27]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
1,3,5-Trinitrobenzene	102	13	64	139
1,3-Dinitrobenzene	103	18	47	158
2,4-Dinitrotoluene	98	12	61	135
2,6-Dinitrotoluene	99	13	60	137
2,4,6-Trinitrotoluene (TNT)	98	15	52	143
2-Amino-4,6-dinitrotoluene[28]	101	17	50	153
2-Nitrotoluene	88	15	43	133
3-Nitrotoluene	90	14	48	132
4-Amino-2,6-dinitrotoluene[28]	104	16	55	154
4-Nitrotoluene	90	14	48	132
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	106	18	51	161
Methyl-2,4,6-trinitrophyenylnitramine (Tetryl)[28]	98	25	22	174
Nitrobenzene	94	15	49	138
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	99	6	81	116

TABLE D-13 LCS CONTROL LIMITS FOR SW-846 METHOD 8330 SOLID MATRIX[29]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
1,3,5-Trinitrobenzene	99	9	73	125
1,3-Dinitrobenzene	102	8	79	126
2,4-Dinitrotoluene	102	7	80	124
2,6-Dinitrotoluene	100	7	78	122
2,4,6-Trinitrotoluene (TNT)	99	14	57	140
2-Amino-4,6-dinitrotoluene	102	7	80	124
2-Nitrotoluene	101	7	80	123
3-Nitrotoluene	100	7	77	122
4-Amino-2,6-dinitrotoluene	101	7	79	124
4-Nitrotoluene	101	8	76	125
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	103	10	72	134
Nitrobenzene	100	8	77	124
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	100	9	74	126

[27] LCS control limits were generated with data using solid phase extraction with acetonitrile only, without removing outliers from the data set (see section D.1 for further explanation).
[28] Provisional limits – LCS-CLs were generated with data from less than four laboratories. Limits may be adjusted in the future as additional data becomes available.
[29] Additional limits for poor performing compounds can be found in section D.5.

TABLE D-14. LCS CONTROL LIMITS FOR SW-846 METHOD 8081A WATER MATRIX[30]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
4,4'-DDD	88	20	27	149
4,4'-DDE	87	18	33	140
4,4'-DDT	92	15	47	138
Aldrin	83	19	27	138
alpha-BHC	94	11	60	128
alpha-Chlordane	93	10	63	123
beta-BHC	96	10	66	126
delta-BHC	91	15	46	136
Dieldrin	95	11	62	129
Endosulfan I[31]	80	10	49	111
Endosulfan II	79	17	28	130
Endosulfan sulfate	96	14	54	137
Endrin	95	13	56	134
Endrin aldehyde	96	14	56	137
Endrin ketone	102	8	77	127
gamma-BHC	82	18	27	137
gamma-Chlordane	94	11	62	126
Heptachlor	87	15	42	131
Heptachlor epoxide	96	11	62	131
Methoxychlor	103	16	56	150

TABLE D-15. LCS CONTROL LIMITS FOR SW-846 METHOD 8081A SOLID MATRIX[32]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
4,4'-DDD	81	18	28	135
4,4'-DDE	97	10	68	126
4,4'-DDT	92	16	45	140
Aldrin	93	16	47	140
alpha-BHC	93	10	62	125
alpha-Chlordane	92	10	63	121
Beta-BHC	95	11	62	127
delta-BHC	94	12	57	130
Dieldrin	96	10	67	125
Endosulfan I	74	20	14	133
Endosulfan II	89	17	37	141
Endosulfan sulfate	99	12	62	135
Endrin	97	12	61	133

[30] LCS control limits are not available for Hexachlorobenzene and Toxaphene, although those compounds do appear on the target analyte list for method 8081A (Table C-8 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

[31] Provisional limits – outlier analyses during the LCS study resulted in LCS-CLs generated with data from less than four laboratories. Limits may be adjusted in the future as additional data becomes available.

[32] LCS control limits are not available for Hexachlorobenzene, Hexachlorocyclopentadiene, and Toxaphene, although these compounds do appear on the target analyte list for method 8081A (Table C-8 in Appendix DoD-C). Sufficient data were not received for those analytes during the LCS study to perform statistically significant analyses. Additional limits for surrogate compounds can be found in section D.6.

TABLE D-15. LCS CONTROL LIMITS FOR SW-846 METHOD 8081A SOLID MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Endrin aldehyde	92	18	37	147
Endrin ketone	100	11	66	134
gamma-BHC	91	11	59	123
gamma-Chlordane	96	10	66	126
Heptachlor	96	15	51	140
Heptachlor epoxide	98	11	66	130
Methoxychlor	100	14	57	143

TABLE D-16. LCS CONTROL LIMITS FOR SW-846 METHOD 8082 WATER MATRIX[33]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	85	20	25	144
Aroclor 1260	87	19	30	145

TABLE D-17. LCS CONTROL LIMITS FOR SW-846 METHOD 8082 SOLID MATRIX[33]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	90	16	41	138
Aroclor 1260	96	12	61	131

TABLE D-18. LCS CONTROL LIMITS FOR SW-846 METHODS 6010 AND 7470A WATER MATRIX

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aluminum	97	5	83	111
Antimony	98	4	86	110
Arsenic	98	4	85	111
Barium	99	4	88	111
Beryllium	99	4	87	111
Cadmium	100	4	87	112
Calcium	98	4	87	110
Chromium	100	4	88	112
Cobalt	99	3	89	108
Copper	99	3	89	109
Iron	102	4	90	113
Lead	99	4	87	111
Magnesium	98	4	88	109
Manganese	100	4	88	112

[33] LCS control limits are not available for Aroclors 1221, 1232, 1242, 1248, 1254, 1268, and 1016/1260, although those compounds do appear on the target analyte list for method 8082 (Table C-9 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

TABLE D-18. LCS CONTROL LIMITS FOR SW-846 METHODS 6010 AND 7470A WATER MATRIX (Continued)

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Mercury	100	5	85	115
Molybdenum	95	5	79	111
Nickel	100	4	87	113
Potassium	98	4	85	111
Selenium	98	6	80	116
Silver	97	5	82	113
Sodium	99	4	87	111
Thallium	97	4	86	109
Vanadium	99	4	88	111
Zinc	100	4	86	113

TABLE D-19. LCS CONTROL LIMITS FOR SW-846 METHODS 6010 AND 7471A SOLID MATRIX

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aluminum	95	5	79	112
Antimony	96	5	82	110
Arsenic	95	4	84	107
Barium	98	3	88	108
Beryllium	99	4	89	110
Cadmium	97	4	83	110
Calcium	97	4	84	109
Chromium	99	5	85	112
Cobalt	98	4	86	110
Copper	97	3	88	106
Iron	100	4	88	113
Lead	95	4	83	107
Magnesium	96	3	87	106
Manganese	97	4	85	109
Mercury	100	6	83	118
Molybdenum	96	5	80	111
Nickel	97	4	86	109
Potassium	96	4	83	108
Selenium	93	4	80	106
Silver	96	7	75	118
Sodium	96	4	82	109
Thallium	94	4	82	107
Vanadium	99	3	89	109
Zinc	95	5	80	110